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Scientific glass.

4145 ① Glass ② Glassblowing
equipments ③ Glassblowing
operations ④ Glass tubing
⑤ ~~Glassblowing~~ seals
⑥ Grinding ⑦ plating
⑧ fractional distillation
⑨ High vacuum ⑩ lathes
⑪ Electrical heaters.

SCIENTIFIC
GLASSBLOWING

E. L. WHEELER
Department of Chemistry, University of California
Los Angeles, California

With a Preface by G. ROSS ROBERTSON

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To EDNA, *my wife,*
and to my children
BILL, BARBARA, DOROTHY, *and* BETTY
for their cheerful sacrifice
of many family activities
during the preparation
of the manuscript

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FOREWORD

From time to time it has been the practice to write a few paragraphs on manipulation of glass tubing before the blast lamp as guidance for the elementary laboratory worker. Such items might appear in a class manual; or perhaps they might be elaborated into a small handbook entitled "glassblowing." More recently, however, the advent of special types of glass, fancy fractionating columns, standard-taper ware, and numerous high-vacuum devices has led the author of this volume to tackle a much more elaborate literary enterprise.

For researchers, particularly in physical chemistry, it has become important to develop more than usual skill in manipulations of modern glass materials and accessories. Such practice has become a substantial and appropriate part of courses in instrumental analysis, and may well be a prescribed program for many doctoral candidates.

Some years ago the undersigned had the official temerity to steal the author of this book from his supposedly permanent position as glassworking genius at Oak Ridge, Tennessee. Since that time the said author has extended and perfected his skill and knowledge in a variety of fields, to our great satisfaction. We are also glad to express our thanks to the Shell Development Company, which had previously demonstrated the validity of its corporate name by "developing" the skill of the author in question, not only in plain glassblowing, but also in small pilot-plant applications of his handiwork. This earlier experience was followed by a period at the Johns Hopkins University where our author not only had to make his gadgets work in actual high-vacuum practice, but also was busy in laboratory instruction in his art. Similar laboratory instruction has been conducted by him from time to time at his newer post in U.C.L.A.

With this background of experience, it is the hope that the readers of this volume may find many suggestive and intriguing hints on laboratory glassworking. Whether you are a "blast-lamp" glassblower, or a crank on "cross-fire" technique, matters not. There is always much to learn.

G. Ross Robertson

June 1957

PREFACE

From my own experiences as a laboratory glassblower during the past twenty-two years and from the observation of operations in other laboratories throughout the country, I came to realize that a glassblower can make of himself either a mere glass manipulator or the research chemist's co-worker. This belief has been further confirmed through talks with other glassblowers. This book was written with the hope that it would broaden the usefulness of other glassblowers whose ambition leads to this co-worker level.

With the research point of view in mind, it seemed appropriate that a book entitled *Scientific Glassblowing* should have sections on subjects not even remotely related to glassblowing, for example, machine shop practices and major chemical processes such as chromatography, extraction, evaporation, and distillation. As a professional glassblower I found knowledge in these subjects both useful and interesting. A glassblower who knows the principles involved in these chemical processes and is familiar with laboratory techniques, such as those of high vacuum, is better able to make constructive suggestions in the designing of new and special apparatus. He is also better able to build the apparatus after it is designed. Furthermore, the glassblower who can make small metal parts or who can design and make the electric heating units which are so often an integral part of glass apparatus, is of special value to his research group.

The burners and glassblowing tools described in Chapter III are those used in modern or American glass blowing in which cross-fire burners are used. The glass tubing is supported on rollers, and blowing into the heated vessel is accomplished by means of a swivel blow tube. This method is given in the procedure for making the basic glass-to-glass seals (Chapter V) and in the advanced techniques of glassblowing (Chapter VI); it is distinctly different from the European method described in most older books on glassblowing. In contrast with the American, the European glassblower uses a single blast lamp burner. No glass support stands are used, and blowing into the vessel is done directly by mouth, without the aid of a blow tube.

Considerable controversy has arisen between glass blowers using the two different styles as to the relative merits of each method. The European style requires a long period of instruction and practice to perfect the basic technique of rotating, heating, and blowing. With sufficient practice the European freehand glassblowing can be developed into a faster method for making standard apparatus, especially on a production basis; however, a distinct advantage of American glassblowing is the ease and speed with which it can be mastered. For precision glassblowing the American method is superior because of the close tolerances which can be maintained and the accuracy with which the work can be lined up.

Included in this book are some topics which have not been covered in similar works. A brief history of glass has been included in the belief that any one sufficiently interested in glassblowing to read a book on this subject would also be interested in the beginning of the use of glass, the early workers, and their primitive technique.

Plating with metal, of great importance in the laboratory, is covered in considerable detail beginning with the chemical plating of silver onto glass for silvered and evacuated glass vessels. Plating of copper and gold by a chemical process is also included as well as the plating of some metals in a vacuum by means of evaporation and sputtering. The solutions and procedures for the electroplating of metals on metal is also included.

In many research laboratories the greater percentage of a glassblower's time is spent in making apparatus to analyze or purify liquids by means of distillation than is spent on apparatus for any one other chemical process. For this reason one chapter is devoted to the subject of distillation, covering the designing, manufacturing, and testing of distillation columns and accessories. Factors which must be taken into consideration in the preparation of a program for precision distillation are listed. Operational procedures for testing columns to determine their theoretical plate or separating efficiency are discussed. Tables and data are also supplied for use in calculating this efficiency and the H. E. T. P. In connection with testing distillation columns, a section on the use and care of a refractometer is included.

A section on electric heaters outlines heating unit bata based largely on Ohm's Law from which the data were calculated. Several tables offer a short cut or guide for the design of heating elements made of Chromel wire and ribbon. The design of coiled units is included and the table "Coil Winding Data" gives the number of feet of wire per inch of close wound coil on arbors from 1/4 to 1/32 inches in diameter.

A chapter covers metal working with a section devoted to the setting up, care and use of a machine lathe. The fundamental lathe operations such as centering the work, work done between centers, work held in a chuck, boring, drilling, and tapping are covered. A description of lathe tools is included along with instructions for their use. Both hard and soft soldering are described as well as spot welding, with two tables showing which metals can be spot welded and approximately the current needed to make successful welds between the different metals.

This book was written with two widely divergent goals in mind. It was to be both a manual for glassblowers in small laboratories (where other service departments and facilities are few, making it desirable for the glassblower to be able to handle a wide variety of work) and as a textbook for beginning glassblowers. To make the book suitable for use as a textbook a great number of illustrations were used. Several types of glass-sealing operations were described and illustrated. Beginners in glassblowing are invited to try the different methods shown before making decisions as to personal preferance in technique and equipment.

I would like to express my appreciation to the members of the faculty at the University of California in Los Angeles for their interest and encouragement during the writing of this book. I am especially indebted to Professor G. Ross Robertson for reading the manuscript during various stages of its preparation and for his valuable, constructive suggestions. I would like to thank Miss Ellen Dunlevy for her help in reading the manuscript, and Mrs. Virginia Dent, Miss Rachel Polk, and my wife who served as typists.

Special mention should be made of R. W. Bemis who drew all the illustrations for this book.

I am grateful to the following individuals and concerns for permission to use some of the material appearing in this book. Dr. H. Sargent, Dr. A. J. Silverman, Dr. Grant Urry, and Dr. W. H. Urry, The American Instrument Company, Bausch & Lomb Optical Company, Bell Telephone Company, Bethlehem Apparatus Company, Corning Glass Works, Fischer Scientific Company, Hoskins Manufacturing Company, Lydon Brothers, Precision Distillation Apparatus Company, Sommer & Maca, South Bend Lathe Works, Stupakoff Ceramic Manufacturing Company, and the United States Rubber Company.

It is also a pleasure to acknowledge the cooperative spirit of the publisher.

E. L. WHEELER

January, 1958

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HISTORY OF GLASS

The origin of the discovery of glass has been lost in the pages of history, but it has been at least 4000 years since glass first appeared to the eyes of an astonished observer as a fire died away. It has been said that, next to the discovery of how to make fires, the discovery of glass was the most important single discovery of the ages.

There were two main steps in the discovery of glass making. First was the discovery that common sand mixed with soda and lime, when heated, can be converted into a new material, very hard and smooth, which we call glass. Second, and equally important, was the discovery of the exact proportions of these materials necessary to obtain transparent glass. No haphazard mixture will do; exact proportions are necessary for the sand, soda, and lime to melt into a crystal clear glass. The first clear glass could well have been an accident, but the second was achieved only after many experiments by chemists whose names were not recorded in the history of glass. So exacting was the work of these chemists that some transparent glass of today is made by the original formula.

THE BEGINNING OF GLASS MANUFACTURE

Pliny,[1] a naturalist and writer of A.D. 23–79, gives an account of the original discovery of the art of glassmaking by the Phoenicians. According to Pliny, "In Syria adjoining Judea and near where the river Belu emptied into the sea was a half mile stretch of sandy beach. The water in this river was unfit for drinking but was held sacred for the observance of certain religious ceremonies. The water was full of slime and deposits, and was quite deep. So deep, in fact, that it was only at very low tides that the little

[1] *The Natural History of Pliny*, translated by J. Boslosk and H. G. Riley, Henery G. Bohm, London, 1857.

stretch of sandy beach became visible. During one of these low tides a ship loaded with soda stopped there for the crew to cook their meal on the sandy beach. There being no stones available to support their cooking utensils, the sailors brought a few lumps of soda from the ship for this purpose, and the heat of the fire fused the soda and sand and produced a stream of transparent liquid that hardened on cooling." This, it was said, was the origin of glass. Again, according to Pliny, this one small spot of sandy beach was for ages the only spot which supplied the material to make glass.

It has been argued by many that such a fire as these sailors used was not hot enough to melt glass. However, Monroe[2] found that a wood fire kept burning 2 hours in the open air developed a temperature of 1200° C. (2200° F.), which is more than sufficient to melt not only a soda-silica glass but also soda-lime-silica glass.

Pliny's story is plausible, but the dating of the oldest pieces of glass indicates that the actual discovery of glass manufacture was made many centuries before this period.

Evidences of the antiquity of glass and glass manufacture are various. The earliest piece of glass bearing a date is a large ball head with the cartouche of Amenhotep (1551–1527 B.C.) which is now in the Ashmolean museum of Oxford. The earliest known glaze is on stone beads of the Badarian age of Egypt, about 2000 B.C. Some evidence indicates that Asia Minor, or more specifically Mesopotamia, was the location of the first glass manufacturing. Excavations in a cemetery of the third dynasty of Ur (2450 B.C.) showed glass beads to be plentiful. A cylinder of light blue glass found by Frankfort[3] possibly dates to the dynasty of Akkad, 2627–2600 B.C. This glass is surprisingly free from striae or inclusions. There is no doubt about the dating of the "Rich Blue Beads" from Deir el Bakri in Egypt, found in the coffin of the little Queen Maït (2196–2172 B.C.). Exploration by Thompson[4] at Nineveh gave considerable information on the art of glassworking. Cuneiform tablets were found which, upon translation, gave the names of various kinds of glasses, their formulas, and directions for making them.

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[2] Monroe, W. L., *Window Glass in the Making*, American Window Glass Company, Pittsburgh, Pennsylvania, 1926.

[3] Frankfort, H., *Iraq Excavations of the Oriental Institute* 1932. University of Chicago Press, Chicago, 1934.

[4] Thompson, R. C., *On the Chemistry of the Ancient Assyrians*, Lizac, London, 1925.

Evidence points also to the manufacture of glass as far back as the sixth century in Persia and in China. A fine example of Persian glass blowing is the Cup of Khosru I (532 B.C.). This bowl was made of colored glass and decorated with gold, with the figure of the king wrought on it. In China, too, glass seems to have been made at about this same period. Until recently it was thought that the early glass in China was imported, but later research by Sarton[5] has shown that glass, probably of native manufacture, was used in China as early as 550 B.C. for decorative purposes.

At first glass was solid in form, but hollow vessels were made later. This involved a laborious process in which a core was built upon a wooden or metal rod and then covered with molten glass bit by bit; the glass used was viscous and not much above its softening temperature. After the vessel was completed, the sand core had to be carefully dug out. About 1200 B.C. another technique was developed, that of pressing glass into open molds. Some of the objects made by these two processes were surprisingly intricate and showed excellent workmanship. The dress of a figured subject sometimes consisted of from 100 to 150 pieces of colored glass pulled into threads no more than a thousandth of an inch thick.

For centuries glass was used chiefly for jewelry, ornaments, and mosaic pavements and glass-covered walls. During the later period of the Roman Empire, the variety of objects made of glass expanded greatly. This rapid expansion both in the use and in the manufacture of glass was in part the result of the invention of glassblowing, which Kisa[6] places as just preceding the Christian Era, or at about 20 B.C. This new method created an industrial revolution and changed a luxury into a necessity. Pompeii, destroyed in A.D. 79, has given evidence of this revolution. In its ruins glass products have been uncovered which show glassmaking had attained a high stage of advancement and that window glass of good quality was made and used.

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[5] Sarton, G., *Introduction to the History of Science* (publication No. 376 Carnegie Institute of Washington). Williams and Wilkins, Baltimore, 1927.

[6] Kisa, A., *Das Glas im Altertum*, pp. 296–299, Leipzig, 1908.

The decline of Rome as the center of glass manufacture came in A.D. 330, when Emperor Constantine moved the glassblowers to Constantinople. So fine was the work of these glassworkers of Constantinople that some ancient specimens once thought to be of precious stone were found later to be of glass. As examples, a blue cup 3 in. in diameter, found at Monza, was at first thought to have been cut from a single sapphire. The so-called emerald table captured at Toledo in the 14th century was believed cut from a single emerald. In addition, the famed "Sacro Catino" of Genoa, a dish considered one of the most sacred and valuable relics of the Christian Era, was found to be glass by a French chemist in 1761.

The Byzantine glassworkers were especially skilled in making colored glasses and mosaics, and during this period stained glass windows first made their appearance. It has been suggested that the art of making stained glass windows originated in Greece about this time. Regardless of its origin, this art was introduced into France, where its use spread rapidly. Adalberon, Bishop of Rheims, used colored glass windows, designed to represent Biblical scenes, when rebuilding the cathedral in A.D. 969–988. This is the earliest record of the use of stained glass windows.

Because of the scarcity of historical records from the third to the ninth century, the story of glass, its uses and achievements, is, however, quite incomplete. During these so-called Dark Ages the glass industry flourished in Islam, until, probably as a direct result of the Crusades and the subsequent decline of the Eastern Empire, glass manufacture moved to Venice. Here it entered a period of growth that made Venice the center of the glass industry. When Saint Mark's Cathedral was built in A.D. 1159, the great demand for glass mosaics established the Venetian glass industry. The entire interior of the building was covered with glass mosaics depicting events of Biblical history. Two hundred and fifty years were required to complete these mosaics. The art of silvering mirrors with mercury was known and used in Venice in 1369, and Venetian mirrors for years were the standard of excellence. The glass industries of Venice flourished until in the seventeenth century there were over 300 factories; however, from that time the decline was rapid—so rapid, in fact, that by the beginning of the nineteenth century only one mosaic factory was still in operation.

GLASS MANUFACTURE IN MODERN PERIODS

With the decline of glass manufacturing in Venice, factories were multiplying all over Europe, and glass was becoming more and more a part of daily life. In 1600 the art of making cut glass ware was developed. The English discovered that coal could be substituted for wood as a fuel for making glass.

During the centuries following the beginning of glassmaking each worker was a master craftsman. Each piece of glass was a handmade creation and the great pride taken by the craftsmen in their work was justified. Outstanding workers marked their pieces with their own initials, and many of these pieces that have come down to us show the excellence and enduring qualities of work well done.

In the latter part of the 19th century a mechanized process for blowing and drawing cylinders 30 in. in diameter and 30 feet long for making window glass was developed. In 1902 Emil Fourcault of Belgium invented the process of drawing flat glass from a tank in a continuous operation. In his operation a sheet of glass was raised vertically between rollers and was annealed by convection currents created by its own cooling. The glass was then cut into suitable lengths.

During this time the chemistry of glass remained virtually the same until toward the last of the 19th century, when a wave of enthusiasm for scientific research swept over Germany and glass became the subject of many experiments. The goal of these German scientists was to discover new glasses for optical instruments. The new glasses that were developed looked like the old glasses but had vastly different characteristics. The old glasses were fragile, expanded greatly under heating, and were unstable towards acids and alkalis. The new glasses were strong, withstood heating, and were only slowly attacked by either acids or alkalis. With these new properties, new uses of glass were introduced, and glass entered fields where it had not been considered practical before.

GLASSMAKING IN AMERICA

The art of manufacturing glass came to America in 1608 when a group of refugee glassworkers settled in Jamestown, Virginia. They made beads which were traded to the Indians for valuable furs.

Among the early American glass makers was Casper Wister, a German immigrant who began by making brass buttons in Philadelphia. In about 1740 he sent to Belgium for four glassblowers and set up a shop in Wisterberg, New Jersey, to make brass buttons and glass. It has been said that now there are fewer than thirty pieces of authenticated Wister glass in existence, although many more are purported to have been made there.

During colonial times, Henry William Steigel made glass history. He built a town called Manaheim, near today's Lancaster Pennsylvania. In it were his glass factory, homes for the workers, schools, and churches. He also built a beautiful home for himself, with hand-carved woodwork, the finest of tapestries, and even a bandstand on the roof. So flamboyant were his ways and so recklessly did he spend his money that he was given the nickname of Baron, but Steigel was no mere playboy. He was sincere in his desire to produce as fine a glass here in America as was produced in Europe. Unfortunately his spending was on such a large scale that he came to financial ruin. Everything he had, including his fine collection of glass pieces, was sold to satisfy his creditors. Today some Steigel items sell for more than his creditors received for his entire collection.

Another of the important early glass manufacturers was Deming Jarvis, who founded the glass company of Sandwich on Cape Cod. Jarvis and his associates made history when in 1827 they developed a pressing device and turned out the first pressed tumbler, thus founding a multimillion dollar industry. This company operated until 1888, when a general strike of glassblowers throughout America closed it down. The Sandwich Company never reopened after the strike was settled.

Previous to this, in 1868, E. Houghton started a glass factory in south central New York. The company was small at first but soon developed a reputation of working special glasses for special purposes. Edison went to this company, the Corning Glass Works, in 1878 when he needed a bulb to fit his new electric light. The glassblowers at Corning, in blowing this first light bulb, started an industry that was eventually to become one of the largest in the world. During the era of railroad expansion another problem solved by this same company was the development of standard colored signal lenses that eliminated the danger of an engineer's misreading his signal color. The railroads had other serious glass problems also involving human life. Hand lanterns were in wide use by trainmen and accidents occurred when trainmen's hand signal lanterns broke from the shock of rain or snow. To overcome this hazard, the Corning Glass Works developed a glass that was proof against sharp changes in temperature. Unfortunately this glass was slowly decomposed by water. Corning also had another glass, widely used for battery jars, which was highly resistant to both water and acid, and Corning immediately set to work to unite the good qualities of both glasses. Although at times the problem seemed hopeless, through this effort the well-known Pyrex brand glass was finally produced.

In 1896 Houghton invented the method for drawing thermometer tubing vertically, and at the present time Corning thermometer tubing is drawn vertically in a twelve-story building.

THE GLASS AGE

According to Silverman[7] the first half of the 20th century experienced such extraordinary development in the manufacture and use of glass that it might appropriately be called the Glass Age. In 1903, Michael J. Owens completed his first workable bottle-making machine. Because of his inventions of many mechanical glassworking machines, Michael Owens has been called the father of mechanized glassworking. Glassmakers say the name of Owens, now incorporated in the title of several leading companies, ranks in the glass industry with those of Goodyear and Whitney in rubber and cotton.

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[7] Silverman, A., *J. Chem. Educ.*, **30**, 32 (1953).

Among the many strange and useful forms in which glass is made are light blocks of glass that can be sawed, cut, or drilled with carpenter's tools. These are made by firing ordinary glass with carbon. The carbon acts with the same leavening effect as yeast during the baking of bread and the finished blocks are a honeycomb of minute cells. A piece the size of 1 pound of ordinary glass weighs little more than 1 ounce. It is waterproof and fireproof, and it is used as insulation in such places as railwaycars, houses, and refrigerators, where its light weight and durability are desirable.

Glass is also made into thread or glass wool. A strand of glass fiber may be 1/15 the diameter of a human hair, but have the tensile strength of steel. The strands are drawn from glass marbles at a speed of more than a mile a minute and a single marble makes a strand that would reach from Washington, D.C., to Wilmington, Delaware. A pound of fiber would go completely around the world. Some of the many uses to which glass fibers are put are: insulating against heat, cold, and electricity, and deadening sound and vibration. Fine fabrics and window draperies are made from glass and may be purchased in department stores of some of the larger cities. One distinct advantage of all glass draperies is the fact that they do not need to be washed or ironed. When they do become soiled, simple immersion in a cleaning solvent and hanging out to dry is all that is necessary.

As Hostetter[8] said, "The imagination is really fired when one considers the many interesting and useful properties of glass. It is as brilliant as a diamond, as fiery as an opal, as colorful as the rainbow, light and delicate as a spider's web, or as huge and massive as a twenty-ton mirror, fragile as an egg shell or as strong as steel. Truly, it can be said that glass is the unusual material; without it we would return to the Dark Ages. With it science and civilization moves on." Today Corning Glass Works alone has over 50,000 formulas for different glasses. The most widely known is Pyrex Brand, Corning No. 7740, and it is in the working of this glass that this book is primarily concerned.

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[8] Hostetter, J. C., *Bull. Am. Ceram. Soc.*, **15**, No. 4 (1936).

II

GLASS AND ITS WORKING CHARACTERISTICS

DEFINITION OF GLASS

Webster's International Dictionary describes glass as an "amorphous substance," usually transparent or translucent, consisting ordinarily of a mixture of silicates, but in some cases of borates, phosphates, etc. Taylor[1] describes glass as a "supercooled liquid that in composition may range from a pure element such as selenium or a pure oxide such as silica, to a complicated mixture of oxides or other compounds."

Glass is also defined as a liquid the rigidity of which is great enough to permit it to be put to certain useful purposes. Glass has no definite melting point but instead has a softening range. Heating glass at even a very low temperature produces some softening, although this softening may not be noticeable to the eye.

COMPOSITION OF GLASS

Silica is the outstanding glass-forming oxide. If it were not so difficult to reach the high temperatures necessary to melt quartz and release the trapped bubbles, and for the glassblower to heat it sufficiently to make seals, silica glass would be the most suitable material for the many uses of glass. Silica has the greatest resistance to attack by water and acid and the lowest coefficient of expansion, and is also relatively free from devitrification.

Fused Quartz

Quartz is one of the many forms of silicon dioxide, the most common of all minerals, and sand is its best known form. Quartz is found as colorless transparent crystals which are practically pure silica and sometimes as large as 20 in. in diameter. Most of the commercial quartz tubing and apparatus we use today is made from Brazilian quartz crystals because of their high purity.

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[1] Taylor, W. C., in a paper given before the Glass Container Association, Montreal, Canada, September, 1928.

According to Devers[2] there are two forms of quartz, sand and rock crystal, each of which is used in the making of functional pieces of fused quartz. The type known as translucent quartz is made from a very pure grade of sand and receives its name from the satin smooth finish caused by the imprisonment of myriads of air bubbles during manufacture. The second type, clear quartz, is made from natural rock crystals, fused in graphite molds under vacuum to bring the air bubbles to the surface of the molten quartz and free them. The rock crystals are molded into solid ingots, from which quartz rod is drawn, and hollow ingots, from which quartz tubing is drawn.

Fused quartz has good mechanical strength; its compression strength is nearly 190,00 p.s.i., and it has a tensile strength of about 7000 p.s.i. Quartz has almost perfect elasticity and gives excellent service as suspension for galvanometer mirrors. Because of its low coefficient of expansion, lower than that of any known material over a wide range, quartz was used by the Bureau of Standards as the material for 10-in. optical flats, believed to be the flattest of any surface in the world. They deviate only 1/50 of a yellow light wavelength, or four ten-millionths of an inch from a perfect plane.

Fused quartz is chemically inert to attack by acids even when hot, except for hydrofluoric acid at all temperatures and phosphoric acid above 150°C. Alkaline solutions attack quarts slowly at room temperatures but more rapidly at higher temperatures.

Commercial Glasses

Because of the difficulty and expense of manufacturing silica glass, other oxides are added to flux the silica and reduce its viscosity. The most widely used flux is soda (equivalent of Na_2O), usually added in the form of sodium carbonate, but according to

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[2] Devers, P. K., *Magazine of Light,* No. 2, 18 (1943).

Taylor[1] its use is limited by the fact that it makes glass more soluble and less resistant to weathering. It raises the coefficient of expansion, making the glass more difficult to work without checking, more difficult to anneal, and less resistant to temperature changes. In other words, it renders glass more brittle, and gives it a surface which is not as tough and scratches more readily. Lime is added to give the glass more chemical resistance and surface hardness. In excess, however, it may lead to crystallization and too quick a setting rate, with the result that the glass does not have the proper working range. Potash may be substituted in part or entirely for soda, and magnesia is sometimes used in place of lime. A small amount of alumina is used in some glasses and increases the chemical stability; however, too much alumina increases the viscosity and makes the glass hard to melt and work.

Table I gives the chemical composition of a number of glasses of interest to the glassworker. Other oxides, such as lead, zinc,

TABLE I. Composition of Some Laboratory and Commercial Glasses

	1	2	3	4	5	6	7	8	9	10	11
SiO_2	80.5	73.0	77.9	71–73.5	61.5	72.68	63.0	72.0	57.3	75.3	68.0
B_2O_3	12.9	16.5	14.1	—	—	—	—	12.0	23.6	7.6	—
Na_2O	3.8	—	4.1	14–17	8.8	13.25	7.6	11.0	1.9	5.7	22.0
K_2O	0.4	4.5	2.3	0–1.5	5.3	—	6.0	—	0.5	0.8	—
CaO	—	—	0.8	5–6	—	12.76	0.3	—	4.7	1.1	7.0
MgO	—	—	—	3.5–4.5	—	0.26	0.2	—	8.7	—	—
Al_2O_3	2.2	—	0.8	—	—	—	—	5.0	3.2	6.2	3.0
Fe_2O_3	—	—	—	.006–0.1	0.8	—	—	—	—		
MnO	—	—	—	—	—	—	0.2	—	—	—	—
PbO	—	6.0	—	—	23.6	—	21.0	—	—	—	—
As_2O_3	—	—	—	—	—	—	0.05	—	—	—	—
BaO	—	—	—	—	—	—	—	—	—	3.5	—

Key: Glass No. 1, Pyrex brand #7740; No. 2, Pyrex brand #7720 (Nonex); No. 3, silex; No. 4, modern lime bulb glass; No. 5, lead glass; No. 6, flat drawn window glass (Libby-Owens process, American); No. 7, American lead bottle glass; No. 8, borosilicate thermometer glass; No. 9, Jena Supermax (German); No. 10, Jena Gerate (German); No. 11, Christmas tree ornament glass.

and barium, are used when special properties are desired, ordinary window glass for example, contains enough iron oxide as an impurity to absorb the ultraviolet rays. If this impurity is removed, the rays will pass. By the deliberate addition of iron oxide the heat rays are stopped, and the resulting glass is used extensively in air-conditioned cars and buildings.

Generally speaking, silicate glass can be divided into four types: lime, lead, borosilicate, and colored glasses.

Colored Glass

Colored glass is divided into two types, one of which is not affected by working conditions and is dependent on chemical composition for its uniformity. The other type, in addition to chemical composition, is dependent on a time-temperature cycle. In general, the colors of the purple-blue-green end of the spectrum are produced by direct coloring oxides not affected by heat treatment. Reds, some yellows, and all opals, on the other hand, are materially affected by heat. Table II gives some of the oxides used in producing colored glass.

TABLE II. Colored Glasses

Coloring agent	Color of glass produced
Cobalt oxide	Blue
Magnesium oxide	Violet
Gold or selenium	Red
Uranium, iron, carbon, or silver	Yellow
Cerric	Brown
Iridium	Black
Copper, chromium, or both	Green
Fluorides or phosphates	White or opal

A detailed study of the composition of glass is given by Morey.[3]

Soft Glass

Because of the high percentage of sodium in soft glass, it is often called soda-glass or lime-soda-glass. Soft glasses also contain potassium, calcium, and varying amounts of aluminum and iron. The high coefficient of expansion of soft glass (nearly three times that of Pyrex glass) makes soft glass less resistant to extreme variations in temperature. It is thus not satisfactory for many types of chemical research apparatus. Because of this lack of thermal shock resistance, soft glass makes an ideal practice material for glassblowing. More care must be exercised in preheating, sealing, and annealing than when Pyrex glass is used.

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[3] Morey, George W., *The Properties of Glass*. Reinhold, New York, 1938.

Pyrex Brand Glass

A low-expansion borosilicate glass known as "Pyrex brand chemical resistant glass No. 7740," was developed by Corning Glass Works in 1915 and is used almost exclusively in the manufacture of laboratory equipment at the present time. "Pyrex" is a registered trademark, owned by the Corning Glass Works, Corning, New York, under which glasses of several different compositions are sold. Glasses which meet certain relative standards of thermal shock resistance, such as glasses No. 7740 and chemical resistants Nos. 7730 and 7070, may be sold under this Pyrex trademark; however, any further reference in this book to Pyrex glass will be to chemical-resistant Pyrex glass No. 7740. When Pyrex glass of another composition is referred to, it will be listed by number. In contrast to soft glass, which is composed of sodium, potassium, calcium, aluminum, and iron, combined with silica, Pyrex contains a large percentage of uncombined silica, a substantial amount of boron, no calcium, and a little aluminum.

Lead Glass

Lead glass enjoys wide use in making tubes for rare gases and fluorescent lights. It is especially adaptable for the glass to metal seals required for this type of work.

In working lead glass a very high oxidizing or "hissing" flame is needed; otherwise the seal will become blackened by the reduced lead liberated. Often this blackening may be cleared up by reheating the seal in a highly oxidizing flame; however, care must be taken when making vacuum seals with lead tubing. Seals should not be started with blackened tubing. Seals made under these conditions have a tendency to leak and once the lead is worked into the glass seal, no amount of additional heating will remove it and correct the leak.

Nonex No. 7720

Nonex 7720 glass is made by Corning Glass Works and is principally used for glass to metal seals. Since Nonex contains lead, the same oxidizing or "hissing" flame used for lead glass is required in heating it.

Vycor Brand Glass No. 7900

Vycor brand glass No. 7900 contains 96 % silica and is produced by a special leaching process developed at Corning Glass Works. The outstanding properties of this glass include the low coefficient of expansion and correspondingly high resistance to heat shock; the high softening point and high chemical resistance make this product extremely useful in the laboratory. This glass can be used continuously at temperatures up to 900°C. or for limited service at higher temperatures, provided it is not in contact with materials that react with or cause devitrification in silica.

Vycor Brand Glass No. 7910

Vycor brand glass No. 7910 contains 96 % silica and is produced especially for purposes requiring high ultraviolet transmission at wavelengths down to 254 mμ. or less. Number 7910 is guaranteed to have a transmission at this wavelength of 70 % for a thickness of 2 mm., but usually transmission is higher. Nordberg[4] states that this values is for observed transmission uncorrected for reflection losses. The maximum possible transmission for zero light absorption in the glass is about 92 %. Both glasses 7900 and 7910 are highly transparent to the near infrared.

Other Vycor Brand Glasses

There are five additional Vycor brand glasses composed of approximately 96 % silica, the differences among these glasses are mainly the finishing techniques to meet a specific end-use requirement. These glasses are:

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[4] Nordberg, M. E., *J. Am. Ceram. Soc.*, **27,** 10 (1944).

No. 7911. A Vycor specially fired for longer life at high operating temperatures. This glass transmits short wavelengths of infrared and has a uniform high transmission of the short wavelength ultraviolet. It also has a notably high electrical resistance and low power loss.

No. 7912. This Vycor is similar to the No. 7911, but transmits ultraviolet light below 185 millimicrons.

No. 7913. This Vycor has the greatest heat resistance of all Vycor glasses, and can be used intermittently at 1300°C. It has good ultraviolet transmission at wavelengths of 365 millimicrons and higher.

No. 7930. The porous unshrunk form of Vycor No. 7900.

No. 7950. A red stained Vycor glass which absorbs most of the visible light from a tungsten filament but transmits infrared radiations.

Lamp Working Properties of Vycor

The lamp working properties of Vycor glass are similar to those of fused quartz. The flame temperature required is much higher than is necessary for working Pyrex glass, but not quite as high as that for silica. For small operations a gas-oxygen flame is satisfactory, but oxygen-hydrogen is necessary for larger operations. As is true with silica, Vycor glass does not flow readily, even at high temperatures, as does ordinary glass. However, because of its resistance to heatshock, the danger of cracking during work is very low. Because of the high temperature used during sealing operations, silica volatilizes and the vapors condense on the cooler portion around the seal to form a white bloom. This can be minimized by limiting the heating to as low a temperature as possible. This bloom, if not allowed to become too heavy, may be removed by heating or by treatment with dilute hydrofluoric acid. Since the temperature at which Vycor is leached is considerably lower than that required for glassblowing, bubbles are usually formed during the sealing. This bubbly appearance does not ordinarily impair the usefulness of the seal except for appearances and clarity. These bubbles may be kept to a minimum by working at as low a temperature as possible.

No alloy has been developed that matches the expansion characteristics of 96 % silica glass. According to Sands,[5] Invar has a low coefficient of expansion but only at relatively low temperatures. In general, it is necessary to use graded seals for introducing metal electrodes or other parts into spherical lamps and tubes.

Corning No. 7280

Corning No. 7280 is a boron-free glass, especially desirable when resistance to alkalis is important. Because of the high expansion coefficient, twice as high as that of Pyrex, this glass should not be used when resistance to thermal shock is desired.

Uranium Glass No. 3321

Uranium glass or the present substitute, Corning No. 3321, is recommended for step seals between Nonex and Pyrex glasses. It is also widely used for making tungsten seals.

Pyrex Glass, Corning No. 7991

Corning No. 7991 can be used for tungsten seals in place of Nonex 7720, the main advantage being that it is a nonlead glass and an oxidizing flame is not required in working. It also has a higher softening point, so that higher baking-out temperatures can be allowed when it is used in vacuum tubes and similar items than are possible with 7720 glass.

Pyrex Brand Ignition Tube Glass No. 172

Since this glass has a tendency to devitrify, it should be worked in a bushy flame that is relatively cooler than that used for glass 7740. A burner in which gas-air and oxygen can be mixed is the most suitable for this work. The glass should be heated in the outer or oxidizing portions of the flame; by working in this softer or cooler flame, devitrification can be prevented, although the working time will be increased.

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[5] Sands, J. W., "Invar, elinvar, and related iron-nickel alloys," *Metals & Alloys*, **3,** 131, 150 (1932).

PHYSICAL PROPERTIES OF GLASS

Table III gives the physical properties of a number of glasses in use today and of special interest to the scientific glassblower.

TABLE III. Physical Properties of Glasses

Glass code No.	Strain point, °C.	Annealing point, °C.	Softening point, °C.	Expansion coefficient, × 10^{-7}	Work point, °C.
7040	450	484	702	47.5	1080
7052	438	475	708	46.0	1115
7060	463	495	690	50.0	
7070	455	490	715	32.0	1110
7280	578	620	870	64.0	
7720 ✓	484	518	755	36.0	1110
7730	507	546	805	36.5	
7740 ✓	515	555	820	32.0	1220
7780	464	498	763	37.0	
7900	820	910	1500	8.0	
7910 ✓	820	910	1500	8.0	
7991	529	565	800	41.0	1175
3321 ✓	497	535	780	40.0	
G–1	389	425	626	90.0	970
G–8	475	510	696	92.0	1000
Silica ✓	1070	1140	1650	5.0	

Working Point

The working point, as shown in Table III, is the temperature at which glass has a viscosity of 10^4 poises. The working point does not constitute a recommendation as to the proper temperatures for sealing, but it is in the range where seals are usually made. It is given to permit the glassblower to compare the approximate sealing temperatures of various glasses.

Annealing Point

The annealing of glass is a problem of great practical importance to the glassblower. When glass apparatus is cooled without special annealing treatment, it is usually strained and subject to breakage. The annealing point shown in Table III is the temperature at which glass will anneal in 15 minutes; at this temperature glass has a viscosity of $10^{13.4}$ poises. Littleton and Roberts[6] determined the annealing point and strain point by a method based on the rate of deformation under torsion at various temperatures. Annealing depends on two factors: time and temperature. As the temperature increases, the time to relieve strain is decreased. The annealing temperature shown in Table IV is one which will relieve strain in a comparatively short time and which is not high enough to cause the glass to change shape under its own weight.

TABLE IV. Oven Annealing of Glass

Wall thickness	Annealing temp., °C.	Soaking time	Initial cooling rate	Strain temp., °C.	Final cooling rate
1 mm.	600	16 sec.	224° C./min.	503	54° C./sec.
2 mm.	592	64 sec.	56° C./min.	503	13.5
3 mm.	587	2 min. 24 sec.	25° C./min.	503	6.2
4 mm.	582	4 min. 16 sec.	14° C./min.	503	3.5
5 mm.	580	7 min.	9° C./min.	503	—
Mixed sizes	560	30 min.	Slow	503	—

Flame Annealing of Glass

Annealing in an oven or furnace is the most satisfactory method, since the entire piece of glass is heated and cooled uniformly and the temperature control is more accurate. Many glassblowers, however, must depend on flame annealing. This consists of heating the worked section in a soft bushy flame (gas-air) until it has been uniformly softened. The glass is at the proper temperature for annealing when it is bathed in a soft orange (sodium) flame. The annealing temperature is gradually decreased by lowering the flame temperature, and this heating is continued until a layer of soot has been deposited from the gassy flame which is used last. The glass should be laid aside to cool in such a manner that the hot glass does not come in contact with a cold table top. Flame annealing requires good technique and judgment on the part of the glassworker, since it is necessary to heat the article uniformly and to avoid overheating, with the resultant softening and deformation of the articles being annealed.

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[6] Littleton, J. T., and Roberts, E. H., *J. Opt. Soc. Amer.*, **4**, 224 (1920).

Some professional glassblowers anneal glass with gas-oxygen flames. Excellent results may be quickly obtained this way by the experienced worker. For the beginner, the danger of either not annealing the glass or of overannealing and causing sag, cannot be emphasized too strongly. When at all possible, the use of gas-air burner for annealing is recommended.

Strain Point

The strain point is the temperature at which glass anneals in about 16 hours, and it may be cooled quickly from this temperature with no damage. At this temperature glass has a viscosity of $10^{14.6}$ poises.

Bloom in Soft Glass

Frequently in soft glass a faint opalescent ring appears a short distance away from the heated area. This can be wiped off at first, but a prolonged heating produces a film which can be removed only by actually fusing the glass.

Devitrification in Old Glass

Old glass or glassware that has been used for chemical reactions often has a tendency to devitrify on being heated. At times, this devitrification occurs before the working temperature is reached. Sometimes this may be cleared up again when the glass is subjected to a higher temperature, but more often heating only makes the devitrification worse. Devitrification is a surface phenomenon and appears to be due to the loss of alkali, either by weathering or by a chemical reaction, leaving an excess of silica which crystallizes out. Germann[7] recommends the following method to remove devitrification in old glass:

1. The glass should be washed with dilute hydrofluoric acid to remove the excess silica, after which the glass can be worked in the flame as if it were new glass.

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[7] Germann, A. F. O., *J. Am. Chem. Soc.*, **43**, **11** **(1921)**.

2. It is claimed that devitrification caused by the volatilization of the alkalis from the glass can be partly remedied by heating in a sodium flame. To do this, the glass should be wrapped in asbestos paper that has first been saturated with a strong salt solution. After being wrapped, the glass is held in a flame and heated.

Devitrification from Overheating

Even Pyrex glass will devitrify if worked too long or at too high a temperature. As a rule, when this kind of devitrification takes place, nothing can be done to remove the visible evidence. Raising the temperature only makes matters worse. In Pyrex this devitrification does not seem to weaken the work. Care should be taken to reduce the time of heating to the minimum and to get the desired results with the first attempt if possible.

Coefficient of Expansion

The rate of expansion of glasses with temperature changes is of utmost importance to the glassblower. The successful joining of glass, either to other types of glass or to other materials, such as metals and ceramics, requires that the materials joined have nearly the same coefficient of expansion. Expansivity may be expressed in several ways but, with reference to glass, the common interpretation is the "mean linear coefficient of expansion," that is, the change in length per unit of length per degree in temperature rise. Table III gives the expansion in centimeters per °C. and shows the average coefficient of expansion at the temperature range 0 to 300° C. This interval was chosen because it indicated resistance to thermal shock; however, the coefficient of expansion of the glass at the temperature at which it is worked is not the same as for the 0 to 300° interval.

Collapsing Temperature of Glass

A series of experiments were conducted by Laubengayer[8] in which each glass was subjected to two tests. In the first, a tube open at both ends was heated in an ordinary electric furnace.

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[8] Laubengayer, A. W., *Ind. Eng. Chem.*, **21**, 174 (1929).

The temperature readings were recorded with a Chromel-alumel thermocouple. The temperature was quickly raised to 300° C. and then raised uniformly at the rate of 3° per minute until the tube collapsed. The temperature at which the first sign of collapsing was noticed is shown in Table V. In test No. 2 the tubes were sealed off at one end and evacuated to 3 mm. pressure, then heated in the same way as in test No. 1. The results of the second tests are also given in Table V.

The Jena Bohemian and Moncrieff glasses listed are combustion tubing.

Laubengayer concluded that to avoid collapsing a tube, it should not be heated to a temperature higher than 50° C. below the figure given in Table V.

TABLE V. The Collapsing Temperature of Glass

Type of glass	Open tube	Evacuated tube
Lime glass	700 °C.	585 °C.
Resistance glass	750 °C.	635 °C.
Pyrex glass 7740	820 °C.	670 °C.
Jena glass	860 °C.	720 °C.
Bohemian combustion	860 °C.	740 °C.
Moncrieff combustion[1]	820° C.	770 °C.

[1] This glass devitrifies readily.

Strength of Glass

Glass is a material of reasonably good strength and if properly handled, hardly deserves its reputation for fragility. Under certain conditions it is as strong as iron. Sullivan [9] has stated that the tensile strength of glass in laboratory measurements ordinarily is about 10,000 p.s.i. This means that, provided the laboratory strength could be maintained in large-scale test, a freight car load weighing 40,000 pounds could be suspended from a glass bar 2 in. square. By a special heat-treating process, the so-called casehardening process, the tensile strength of glass can be brought near that of wrought iron, which is about 50,000 p.s.i.

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[9] Sullivan, E. C., "The many sidelines of glass," *Ind. Eng. Chem.*, **21**, 277 (1929).

The resistance to crushing pressure is also very great. Again, provided that laboratory strength held good in large-scale operations, 350 tons balanced on a 2-in. cube of the strongest glass would be just sufficient to crush it. The compression strength of glass is greater than that of ordinary building materials, such as concrete, brick, or granite. Even cast iron yields under three-fourths the pressure required to crush glass. In general, glass maintains its shape under pressures that cause metal to flatten out like putty.

Bursting Pressure of Pyrex

The formula for calculating the bursting pressure of Pyrex tubes and cylinders as recommended by the Corning Glass Works[10] is Lamé's formula without modification. This is as follows:

$$P_e = \frac{-1 + (d/D)^2}{[1 + (d/D)^2]\,p}$$

where d = inside diameter of the tube;
D = outside diameter of the tube;
p = internal pressure in pounds per square inch;
P_e = a commonly chosen value between 800 and 1500 p.s.i.

The choice of values used naturally depends upon the conditions under which the tubing is to be used, as well as the temperature.

The P_e as specified is for thoroughly annealed glassware.

The following is a general rule which may be applied to Pyrex:

Bores up to 0.20 in. maximum stress 1200 p.s.i.
Bores up to 0.75 in. maximum stress 1000 p.s.i.
Bores over 0.75 in. maximum stress 900 p.s.i.

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[10] E. L. Jolley, private communication.

III

GLASSBLOWING EQUIPMENT

INTRODUCTION

Much of the standard equipment used by glassblowers is made commercially and may be purchased at scientific supply houses. This category includes many of the different types of burners, rollers, supports, flask holders, shaping tools, and even polariscopes. A list of firms supplying this equipment is included as an appendix to this Chapter. The author makes no claim that the firms designated are the sole suppliers of each item or that they have the best in each case, merely that they do supply a designated item and that the item can be used satisfactorily.

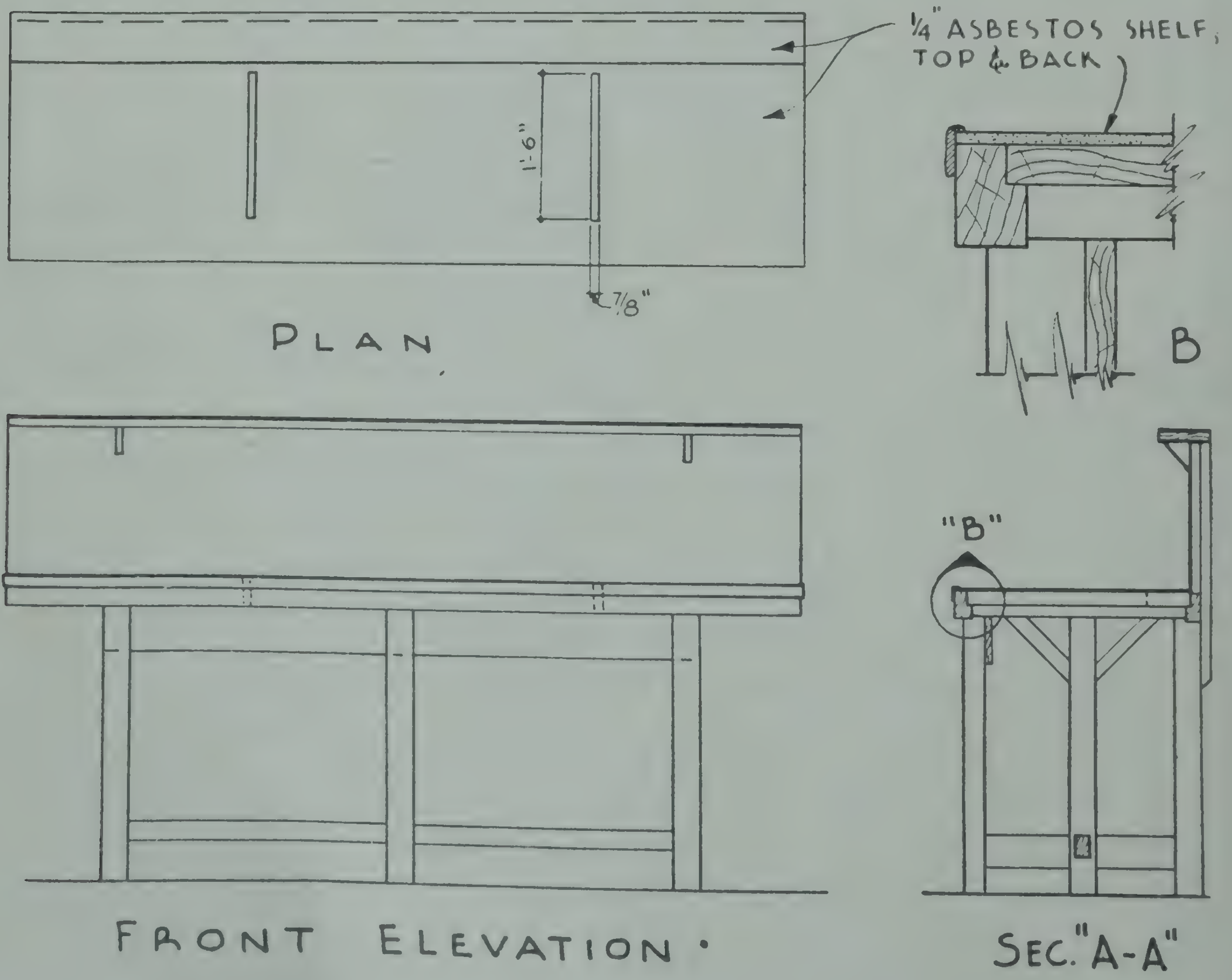


Figure 1A. Drawing of glassblowing bench.

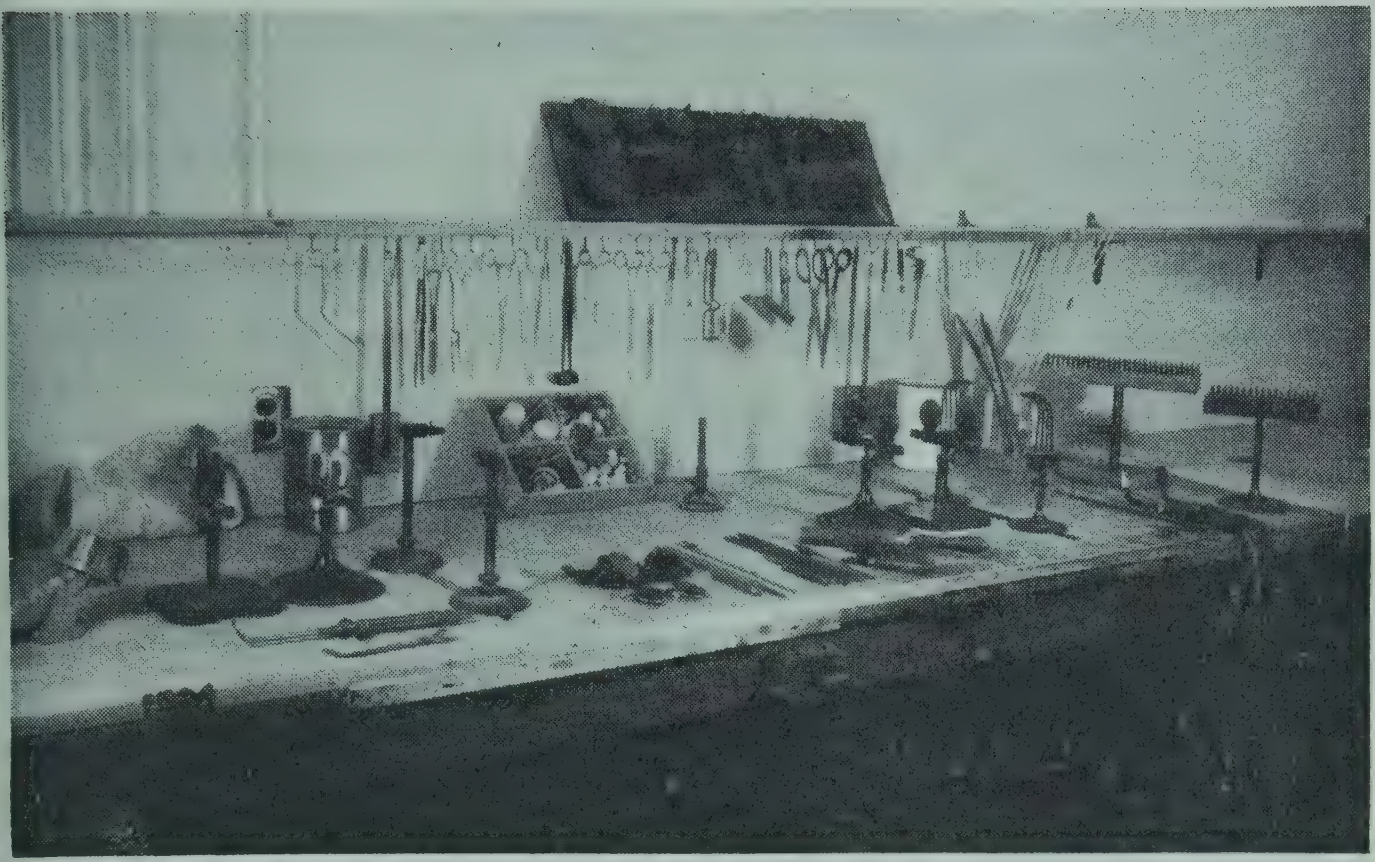
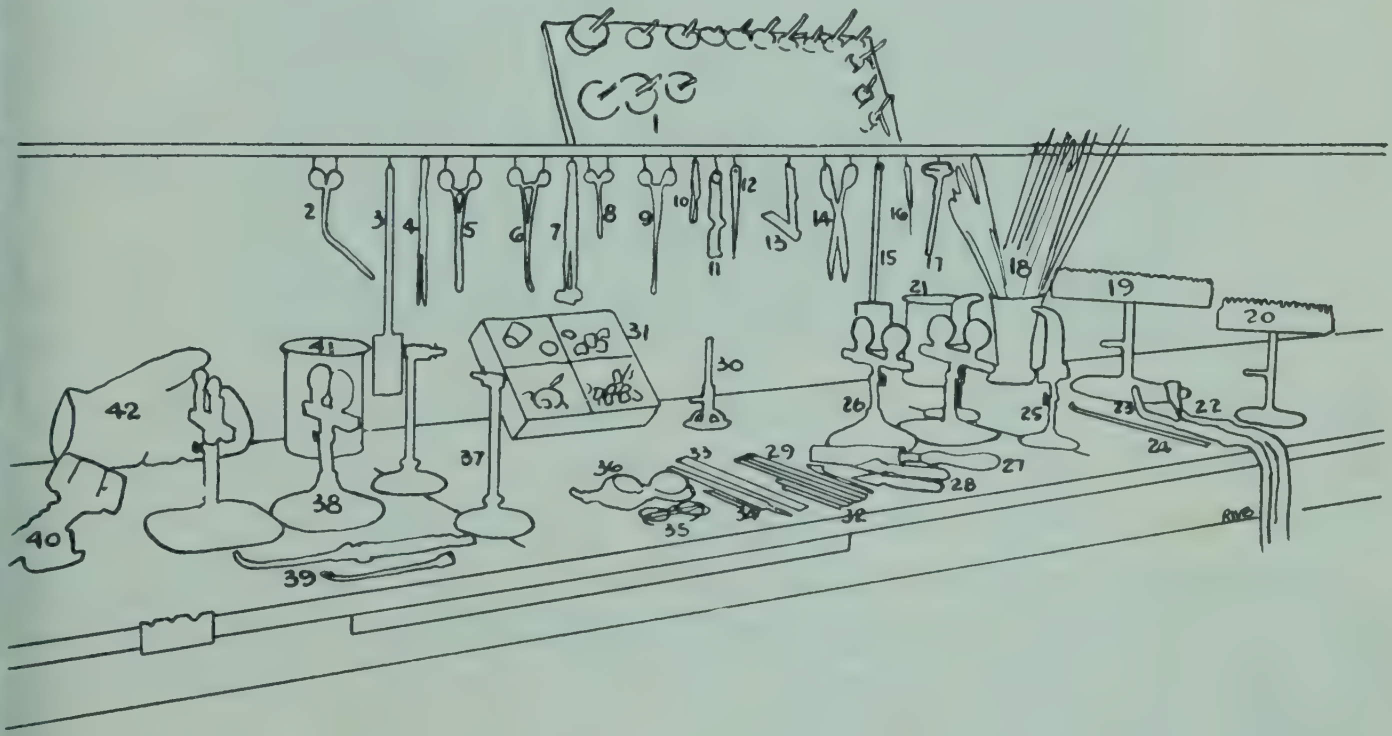


Figure 1B. Photograph of glassblowing bench.



1. Board for rubber stoppers
2. Special forceps
3. Small carbon paddle
4.—10. Assortment of forceps
11. Flint gas lighter
12. Tapered glass tubing gage
13. Jig for setting angles of apparatus
14. Scissors
15. Large carbon paddle
16. Tungsten needle in pin vise
17. Calipers (metric and inches)
18. Stock of glass patch rods
19.—20. Ribbon burners
21. Water pot
22. National gas-air burner
23. National gas-oxygen burner
24. Assortment of National gas-oxygen burner tips
25. Four-tip crossfires
26. Glass support stands (rollers)
27. Carbon hexagonal shaper
28. Brass shapers
29. Carbon rods
30. Modified Bunsen burner
31. Box of corks and rubber policemen
32. Scripto glass writing pencil
33. Ruler (1 foot)
34. File for cutting glass
35. Glassblower's goggles
36. Goggles for quartz blowing
37. Single-tip crossfires
38. Second set of glass supports
39. Victor welding torch
40. Litton annealing burner (gas and air)
41. Second water pot
42. Asbestos mittens

PERMANENT EQUIPMENT

Glassblowing Bench

If any appreciable amount of glassblowing is to be done, it will pay to arrange a special work bench. The work bench should be located so that the glassblower has sufficient room at each end for freedom in handling long pieces of tubing. An important consideration is ventilation. It is not as necessary that the room be air-conditioned as that it be free from drafts. A convenient bench is shown in Figure 1A and 1B. This bench is 30 in. wide by 96 in. long, with slots cut in the top for two sets of cross fires. For workers using blast lamps, these slots are not necessary. Most professional glassblowers, working 7 to 8 hours a day, do their work sitting down. This sitting position allow's the worker's elbows to rest on the table, thus helping to steady the glass when in the flame. The comfort of the worker has a decided influence on the quality of his work, especially in the case of a beginner, who often handicaps himself by working in uncomfortable and strained positions. Thirty-one inches is the customary height of glassblowing benches. For those preferring to stand while working, a height of about 40 in. is satisfactory. The top of the bench should be covered, preferably with 3/8 in. asbestos-cement board, although asbestos paper or masonite is usable. Ideally, gas, air, and oxygen are piped to the bench into a manifold and to valves located at the front of the bench. These valves are within easy reach of the worker, preferably to the right of each pair of burners. A slight modification of this plan is required when foot pedals are to be used (see Figure 2). If a house vacuum line is available, a vacuum outlet on the front of the bench will be found very convenient on special occasions.

Pressure Regulator

A pressure regulator for the oxygen tank is essential if the worker plans to regulate his burners by the valves on the bench. A pressure of 8 to 12 pounds is satisfactory. When oxygen is piped into the room from another location, a regulator at the bench, in addition to the one on the oxygen tank, is advisable. Under these conditions the oxygen is brought into the room under higher (50 pounds) pressure and reduced to 8 to 12 pounds at the bench. If more than one glassblowing station is planned, each station should have separate secondary regulators. For small installations where no pressure regulator is available, the flow of oxygen to the burners may be regulated with the tank valves; in this case the burner valves should be open at all times.

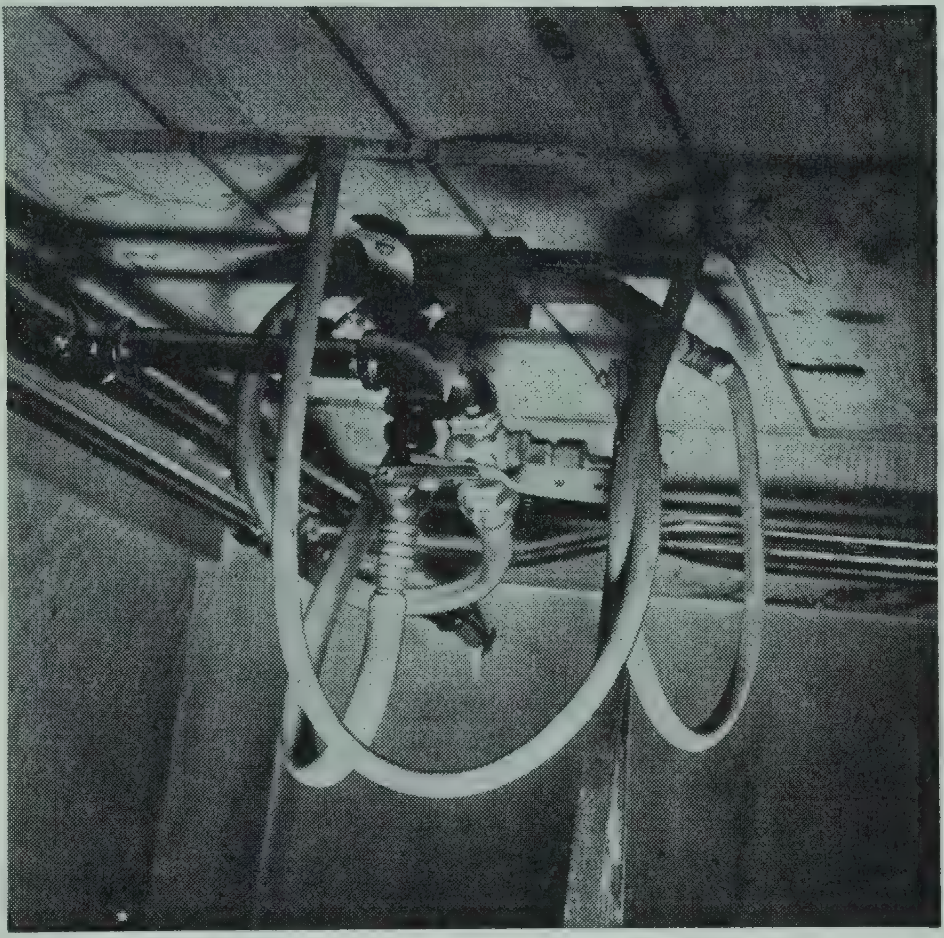


Figure 2. Photograph showing arrangement of piping for foot pedals.

Glass Storage Bins

A cabinet for storing the glass, although not essential, is a convenience. Provisions should be made for storing a wide variety of sizes, but the quantity in each size need not be large. Either horizontal or sloping racks may be used. The ideal cabinet is enclosed to protect the tubing from dust.

Glassblower's Goggles

Goggles are a great help in protecting the eyes from the intense sodium glare of the heated glass. These are made from didymium glass, made by Corning Glass Company. In this glass, a combination of the oxides of two rare earths, neodymium and praseodymium, absorbs the light in the region of the sodium D line. This absorption makes the heated glass clearly visible through the flame. If the lenses of the goggles are left thick, complete absorption of this sodium is obtained. However, a lens 3½ mm. thick gives ample protection and is much more comforable to wear.

Goggles for Blowing Quartz

Because of the intense light given off by quartz when it is heated to the softening point, very dark glasses should be worn to protect the eyes. Welder's goggles may be used and a pair with dark lenses should be chosen.

Glass-Marking Pencil

A grease pencil that writes easily on glass is a necessity. It must also mark the glass enough to remain when heated, yet not burn into the glass so as to leave a large discolored spot when the apparatus is cooled. A Scripto pencil with China-Marking Lead G-920 is satisfactory.

Diamond Pencil

A diamond pencil is useful for marking bottles before cutting with a hot wire, and for many other marking jobs that arise from time to time.

Polariscope

For those workers desiring to make their own equipment, Figure 3A shows a polariscope that can be made easily and inexpensively. The polaroid sheets may be purchased and mounted between plates of glass to form the front and eye piece of the instrument. Several excellent polariscopes may be purchased from glass supply houses.

Figure 3B shows a portable polariscope that may be made from a Boy Scout flashlight. This can be operated with flashlight batteries or remodeled to connect directly to the 110-volt line. A portable polariscope is offered by some supply houses.

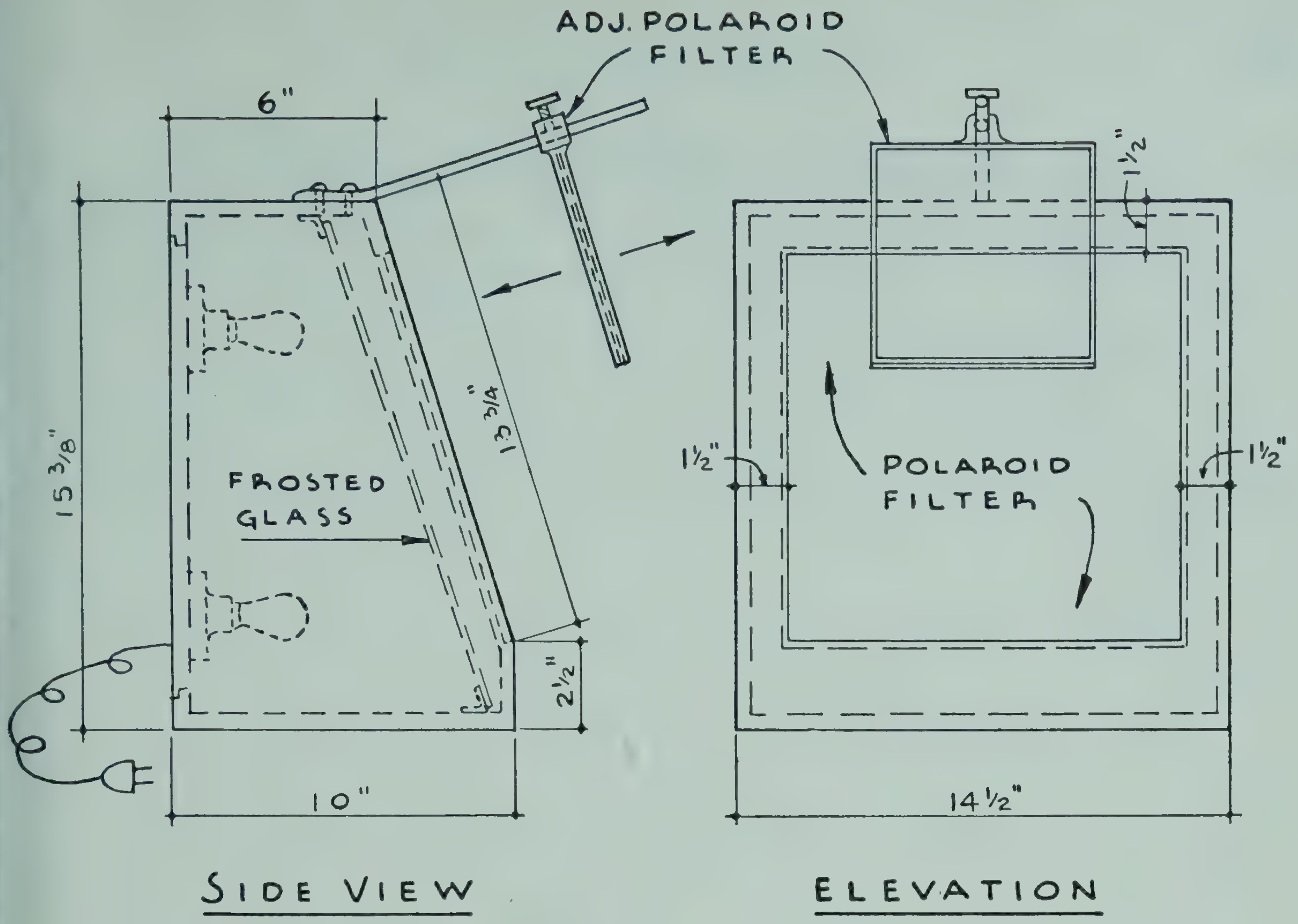

ADJ. POLAROID FILTER
6"
15 3/8"
13 3/4"
FROSTED GLASS
2½"
10"
1½"
1½"
1½"
POLAROID FILTER
14½"
SIDE VIEW
ELEVATION

Figure 3A. Bench polariscope.

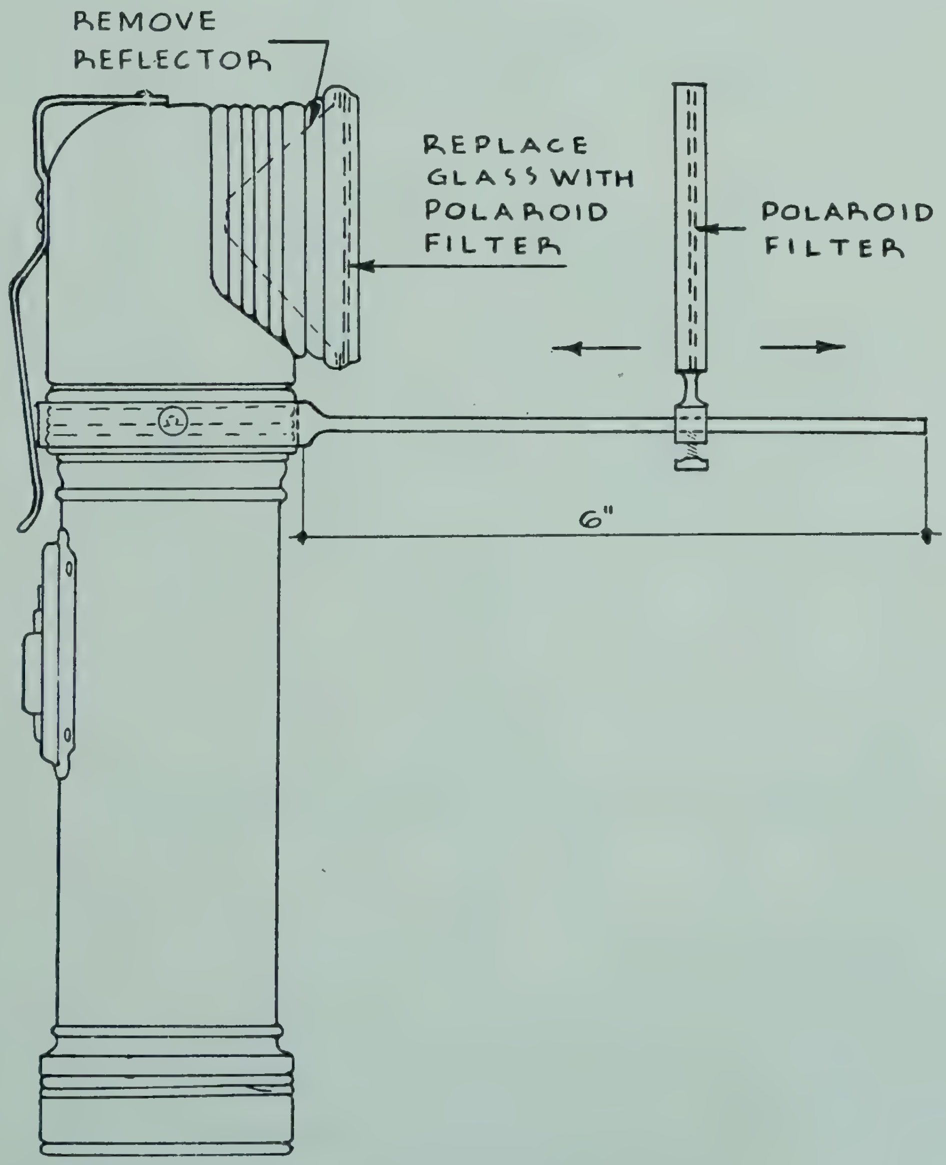

REMOVE REFLECTOR
REPLACE GLASS WITH POLAROID FILTER
POLAROID FILTER
6"

Figure 3B. Portable polariscope.

Although a polariscope is not absolutely essential in a glassblowing shop, it is a useful piece of equipment, especially if no annealing oven is available. In use the polariscope is adjusted with the two pieces of polarized glass set at 90° to each other, thus cutting out the light. In viewing glass, any strain shows as a light spot and its location, size and intensity are ascertained. When necessary, the glass is more thoroughly annealed to prevent cracking.

Bench-Type Glassworking Rollers

No glassblowing bench is complete without a pair of rollers or glass supports, and it is preferable to have one pair for each set of burners that are stationed permanently on the bench. For work with small-diameter tubing, one roller is sufficient, but for larger tubing two rollers are necessary. When four rollers are lined up, two on each side of the burner, long lengths of tubing may be sealed together with considerably more ease than when the tubing is supported in the hands alone. Figure 4 gives the

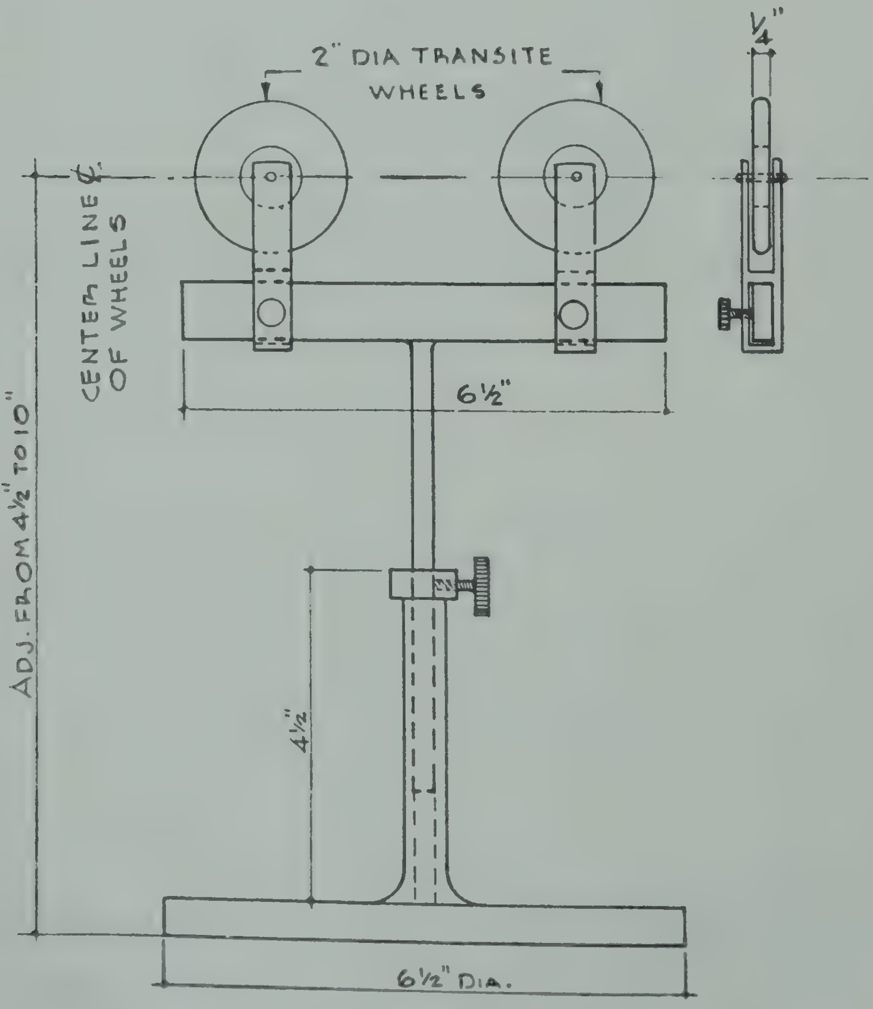


Figure 4. Bench-type rollers.

necessary information for building a set of rollers with ball-bearing wheels. Many types of commercial rollers are available, some quite similar to these described.

Transite Support Stand

A simple glass support may be made from Transite following the description given in Figure 5. These supports may be used satisfactorily in place of ball-bearing rollers during actual glassblowing operations involving rotating. In addition, they are very handy for supporting glass while it is cooling.

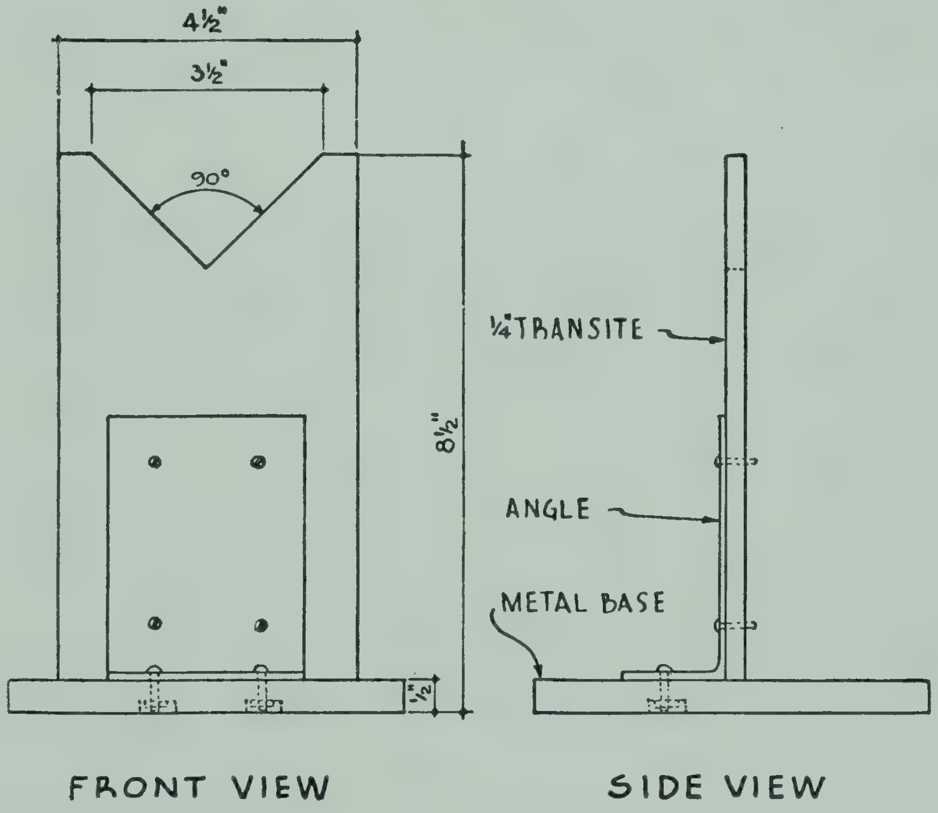


Figure 5. Transite support stand.

MISCELLANEOUS EQUIPMENT

Support for Hot Glass

A convenient rest for pieces of hot glass to prevent their coming in contact with the cold bench top may be made from a notched length of wood (2 × 4 in.). A more elaborate support provides different sizes of pipe and rod to accommodate different diameters of glass tubing.

File

If regular glassblowing cutting files are not available, flat machinist files are satisfactory. Small triangular files are used by many glassblowers; the flat file, however, is advantageous because it allows the worker to grind or resharpen the file on a grinder when the cutting edge becomes dull.

Glass-Cutting Knife

Some glassworkers prefer glass-cutting knives for cutting glass, since these give a slightly finer scratch mark than a file. These are usually made with either a Carbaloy cutting edge or a tungsten carbide edge. Either may be sharpened by lapping the edge on a glass-grinding wheel.

Asbestos Tape

Asbestos paper is a necessity in a glassblowing shop, and it is doubly useful if purchased in the form of tape. These rolls of tape come in two widths (1 in. and 2 in.) and two thicknesses (0.011 in. and 0.019 in.). Asbestos tape is used for supporting inner tubes during fabrication. Construction must be such that the asbestos may be removed before the apparatus is completed. Asbestos tape is also useful for closing open ends of tubing that will be heated while the glassblowing is in progress. Stopcock plugs should be removed and replaced with a roll of asbestos tape while the stopcocks are being sealed to other glass apparatus. Warning: Since the filler used in the manufacture of asbestos paper will burn, it is advisable to keep a supply of preburned tape on hand for use in places that will be heated during the glassblowing operation. Smoke given out during excessive heating will burn into the glass if allowed to collect at the point where the seal is being made. This deposit can be removed by blowing air through the apparatus while at the same time heating the glass with a gas-oxygen flame, provided the glass has not been heated to its working temperature previous to passing the air through the apparatus. If the air treatment is not started soon enough and the contamination is allowed to burn into the surface of the glass, it will be impossible to remove.

Asbestos Cloth Tape

In situations where too much asbestos paper would be required, the thicker cloth tape is advantageous. It is also wise to use this as a covering for completed seals to protect them from flame splash during subsequent glassblowing operations.

Flask Holders

Small flasks may be held and rotated by sealing a piece of glass rod to the bottom. This handle is removed upon completion of the work. Bent wire holders may be fashioned, but, for ease in use and for producing accurate and professional work, a set of flask holders is necessary. With these, many different sizes and even shapes of flasks may be supported and rotated comfortably.

Calipers

One of the most widely used pieces of equipment in a glassblowing shop is the caliper. A popular model of inside and outside caliper has graduation reading from 1 to 120 mm. and a vernier which reads to 0.1 mm.

Glass Tubing Tapered Gage

A tapered gage is especially useful for measuring capillary tubing. A standard commercial item, it is calibrated to read direct to 0.1 mm., in the range of 1 mm. to 15 mm.

Calipers Not Calibrated

For measuring larger glass tubing or distances which cannot be measured with the vernier calipers, inside and outside calipers can be extremely useful.

Reaming Tools

Reaming tools are usually made from flat brass and are in the form of a triangle and fitted with a handle. The thickness of the brass varies from 1/16 to 1/8 in., depending on the length of the tool, which may range from 1 to 4 in. Both sharp and blunt tools are useful. These metal reamers are less likely to stick to hot glass, and will produce a better finish if they are given a coating of Aquadag before using. A satisfactory Aquadag coating can be achieved by repeatedly heating the tool and immersing it in a saturated solution of Aquadag. Once achieved, this coating is kept intact by the continued immersion of the tool in Aquadag after each heating operation.

Carbon Reaming Tools

Glassblower's hexagonal shapers may be purchased having the standard 1—10 taper. These are particularly suitable for work involving repairing of standard taper ($) joints prior to regrinding as well as for forming rubber stopper connections.

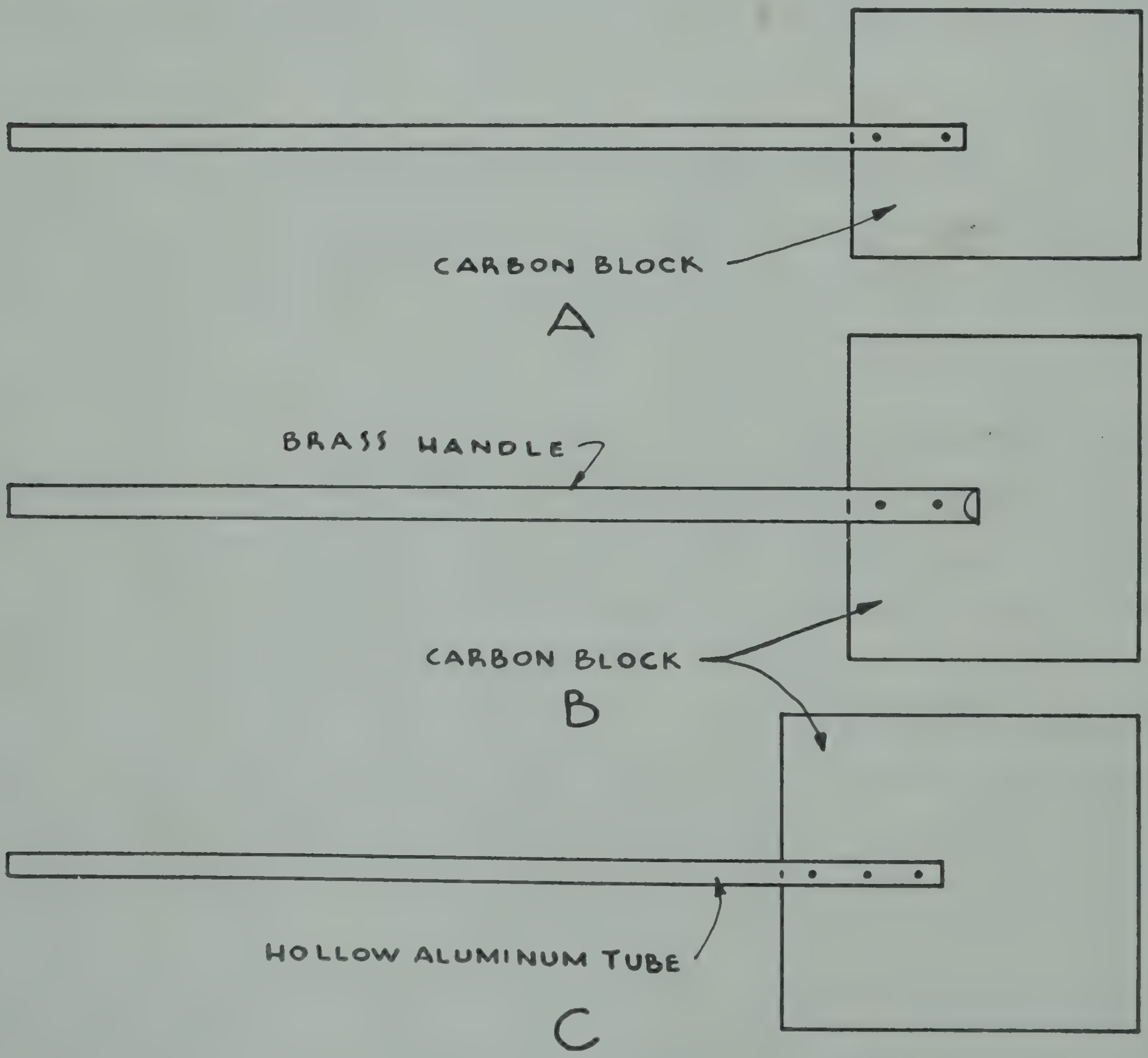


Figure 6. Flat carbon shapers.

Carbon Paddles

A satisfactory carbon paddle may be made from ½ in. thick pieces of carbon plate, as shown in Figure 6. Two sizes are recommended, one 2×4 in. and the other 3×4 in. A 14 in. piece of ⅛×½ in. brass stock makes a suitable handle. Ordinarily the paddle will not be in contact with the hot glass long enough to become too hot to hold. If it does, an insulated handle may be made by wrapping the brass with asbestos paper that has been moistened with sodium silicate.

Carbon Rods

In place of brass flaring tools carbon rods of different sizes may be used. Special fine-grain electrode carbon rods do not mark the glass or conduct heat as quickly as regular carbon reamers. These are available from ⅛ to 1 in. in diameter, in gradations of ⅛ in.

Pencil Sharpener

A standard office pencil sharpener is very useful for grinding sharp points on carbon rods used as flaring tools. Most pencil sharpeners are adjustable to accomodate pencils from ¼ to ½ in. in diameter. This range covers the most widely used diameters of carbon rods. Points may also be ground on rods smaller than ¼ in. if the rod is carefully centered in the smaller hole of the sharpener and held there rigidly while being sharpened.

Forceps

The glassworker will find a pair of 10 or 12 in. biological specimen forceps invaluable on many occasions, both for holding objects and for pulling heated glass. If the forceps are to be used in contact with hot glass for any length of time, it is advisable to obtain regular glassblowing forceps that have a heat-resistant vulcanite handle. A second pair of biological specimen forceps with a flat 1 in. square piece of steel welded to each tip is useful for flattening or shaping hot glass.

Tungsten Needle

A piece of tungsten wire held in a pin vise is very useful for drilling small holes in Pyrex glass. (See p. 154.) It can also be used in repairing small cracks and holes in glass.

Rubber Policemen

Policemen are used for closing one end of tubing preparatory to making a seal. They can be obtained commercially or made by cutting rubber tubing into 2 to 3 in. lengths and closing one end with a small cork. It is helpful to have several sizes on hand, depending on the diameter of the tubing to be used.

Asbestos Mittens or Gloves

The uses to which asbestos gloves can be put are too numerous to mention. They will, however, prove their worth many times over and should be included in any glassblowing shop.

Blow Tube

A length of light ($^1/_8 \times {}^1/_{32}$ in.) rubber tubing fitted with a glass mouthpiece, or preferably with a mouthpiece made from the stem of a pipe, makes an excellent blow tube. If glass is used for the mouthpiece, it should be thickened to prevent the worker from shattering the glass while it is in his mouth.

Swivel

A blow tube is not complete unless it is fitted with a swivel to prevent it from becoming twisted as the glass is rotated.

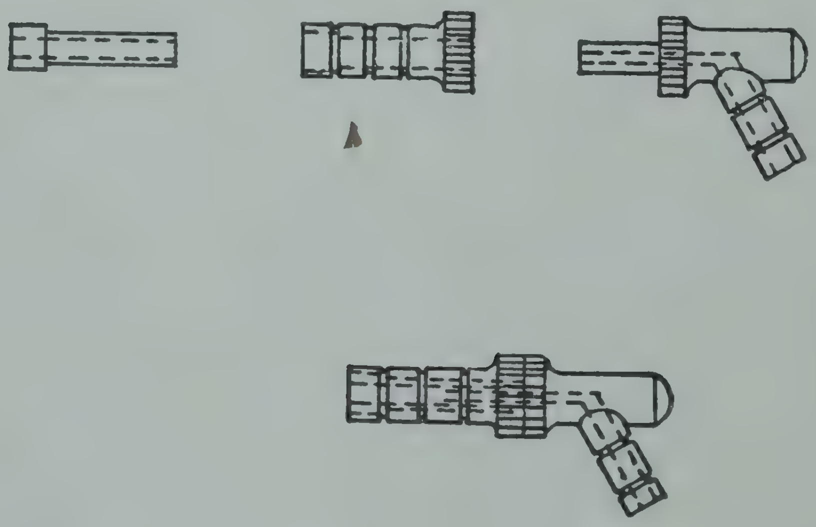


Figure 7. Swivel.

Figure 7 shows an exploded view of a satisfactory swivel. Commercial ones are also obtainable.

Water Pot

At the back of the bench and to one side of each set of burners, it is convenient to have a water pot. This may be made from the bottom half of a bottle or beaker; however, a metal pot is more satisfactory. Scraps of hot glass may be deposited in this water pot, and a fresh clean end to a Pyrex rod can be obtained by immersing the hot rod in the water.

Corks

Corks are necessary for closing the open ends of glass tubing and a box holding an assortment of many sizes should be located conveniently at the back of the bench.

Rubber Stoppers

A board should be prepared for holding at least two stoppers of each size. These should have a glass tube inserted through them for use as a handle and for attaching the blow tube. In some sizes, a stopper with a side arm on the glass handle will be an added convenience. The board should hold stoppers ranging in size from 00 to 12. If larger tubing is to be used, sizes 13, 14, and 15 should be added.

Motorized Rollers

An electrically driven roller is useful during preheating and even for heating during annealing and while cooling the glass. With these rollers the glassblower can do other work while preheating or annealing a second piece of tubing.

Cutting Jig

This handy little cutting jig can be made from a scrap of $^1/_{16}$ in. brass and fastened to the bench with two screws (see Figure 1B, lower left-hand corner). In use, the glass tubing to be cut is scratched with a file and placed in the jig with the file mark up. A sharp tap on the end with a file will readily break the glass tube.

Glass Handles, $

When the glassblowing requirements include sealing joints to apparatus, a method of holding the joints is necessary. A set of prepared $ handles of the various sizes is the answer to this. Most glassblowers prefer to make and align their own handles, however. Commercial handles may also be purchased. The commercial handles have asbestos tape cemented over the ground section to prevent it from freezing or sticking to the joint being held.

Box for Storing Short Lengths of Tubing

It is convenient to have a storage place for short lengths of smaller diameter tubing (up to 35 mm.). Any size box that will fit in the available space will do. A box 6 in. wide by 15 in. long by 6 in. high is satisfactory. It is helpful to have this divided into four to six compartments so that some separation of the various diameters can be made.

Electrically Heated Hot Wire

In Figure 8 of Chapter IV, page 59, is shown the diagram for constructing an electrically heated hot wire for cutting glass. This assembly is designed for direct control of both the heated wire and cold running water. Portable commercial hot wire units may be purchased, but these require the manual addition of cold water necessary for creating a crack in Pyrex glass.

Copper Screen Supports

In situations where it is to the glassblower's advantage to leave the internal supports in until the apparatus is completed, asbestos tape may be replaced with ordinary brass window screening. Screening for this purpose should be purchased in lengths of 15 to 20 feet and cut in strips 2 in. wide. These should be passed quickly through a Bunsen burner to remove the preservative sprayed on the screen and to anneal the metal and remove the spring. After this burning treatment, the copper strips may be wrapped and stored until needed. In most cases, strips 1 in. wide are satisfactory and these can be easily cut from the annealed wider strips. This copper support may be left in the apparatus during oven annealing; however, with apparatus that is to be silvered it is advisable to remove the copper before annealing to prevent the copper from discoloring the glass at that point.

The copper is removed by dissolving in nitric acid.

GLASSBLOWING BURNERS AND FLAMES

The size, variety, and complexity of the burners is regulated by the type of glass to be worked, as well as by the size and intricacy of the glassblowing to be attempted. Table I gives the temperatures of different flames.

TABLE I. Temperatures of Flames

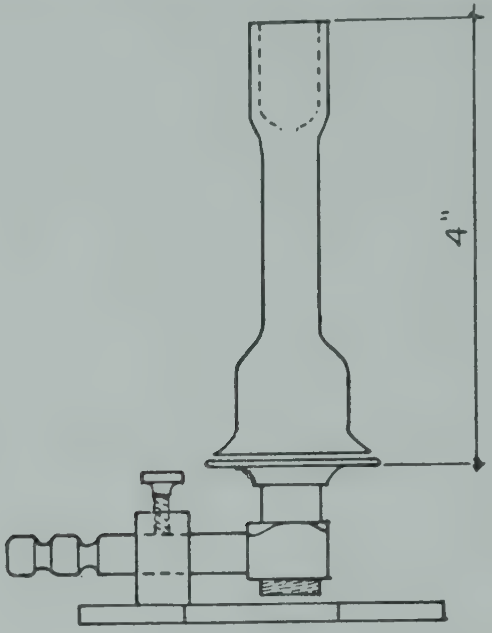
Gases used	Temperature, °C.
Natural gas and air	1871
Manufactured gas and air	2200
Natural gas and oxygen	2260
Manufactured gas and oxygen	2500
Hydrogen and oxygen	2900
Acetylene and oxygen	3000
Acetylene and air	2458

Bunsen Burner

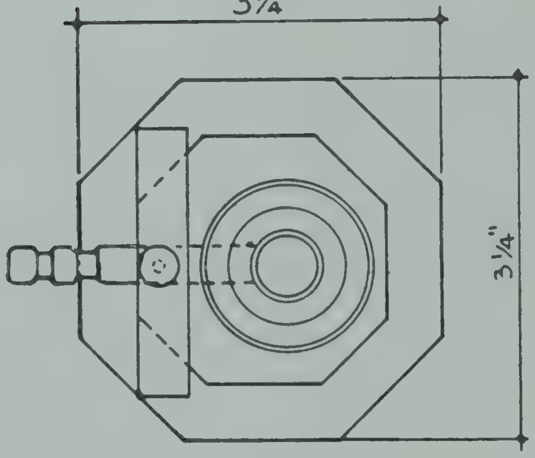
A Bunsen burner is the most common laboratory burner. For simple glassblowing with soft glass, this burner is quite satisfactory. For glassblowers who work with Pyrex glass, a Bunsen burner is primarily used for preheating the glass in preparation for the actual glass seal and for keeping the glass hot while work is in progress. A Bunsen burner is also used on many glassblowing benches as a pilot light when other burners blow out. Figure 8 shows a modified Bunsen burner that can be tilted and focused at the work from many angles. It can be used in many places where a standard Bunsen burner will not fit.

National Hand Torch

For small work and for work on vacuum equipment built in place, a hand torch is invaluable. These burners are equipped with an oxygen tip, and two gas-air tips that can be used for annealing small pieces of glass. A complete set of oxygen tips from size 0 to size 5 is available. Some glassblowers prefer to have two hand torches hooked up to their benches permanently, one for an oxygen-gas flame and the other as an air-gas annealing burner.



ELEVATION



PLAN

Figure 8. Modified Bunsen burner.

Buffalo Blowpipe

This hand torch is similar to a torch known by some glassblowers as a dental torch. It is for use with oxygen and gas and is especially useful if one torch is to be used for both glassblowing and annealing. When this torch is held in the hand, a light pressure will change the flame from a sharp oxygen tip to a full-size brush flame. A regulating nut is provided so that the flame may be changed from one variety to another and back again without readjustment of the gas and oxygen valves.

Victor Welding Torch

The light weight and slender tip of this torch make it especially useful when performing glassblowing in tight or crowded places. It comes with interchangeable tips ranging in sizes from 0 to 3.

Litton Annealing Burner

The author has found that with this large annealing burner, large pieces of glassware can be annealed evenly. It is especially effective used in conjunction with a glass lathe. A similar burner is made by the Western Scientific Company.

Single-Tip Crossfires

These burners are designed for working small glass tubing and for use in places where a sharp hot flame is desired. The gas and oxygen mix at the tip of the burner, eliminating the premixer used with multiple-tip crossfires. The burners shown in Figure 9 are not made commercially, but if a machine shop is

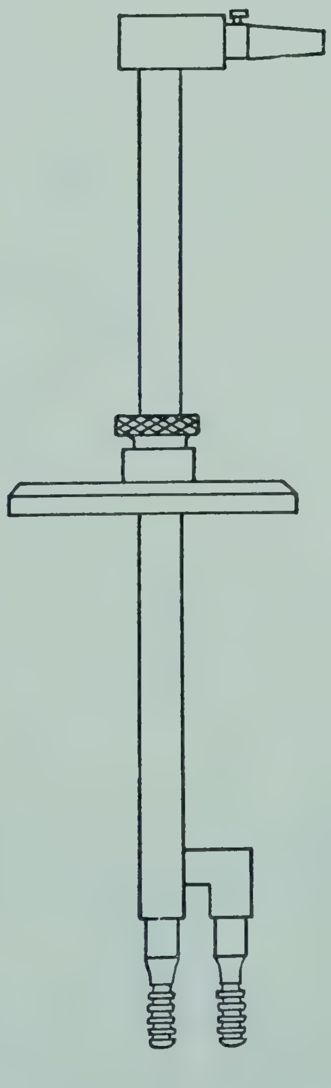


Figure 9. Single-tip crossfires.

available, they may be made according to the drawing. These single-tip burners may be used in pairs as crossfires or singly, as the occasion demands.

Premixer for Gas-Oxygen Burner

Burners that have no mixing chamber in the burner require a premixer, as shown in Figure 10A. The gas and oxygen are

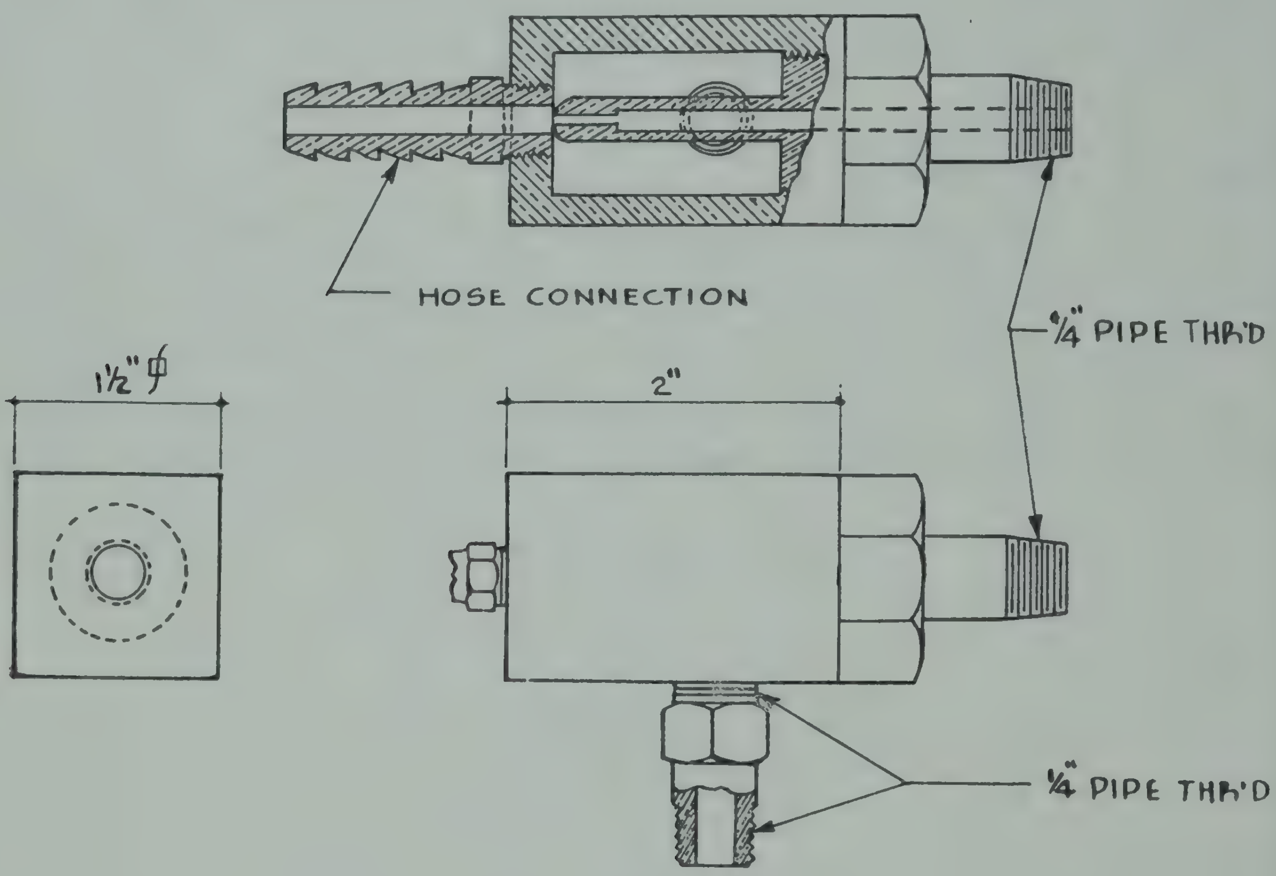

HOSE CONNECTION
¼" PIPE THR'D
1½" ϕ
2"
¼" PIPE THR'D

Figure 10A. Gas-O$_2$ mixer.

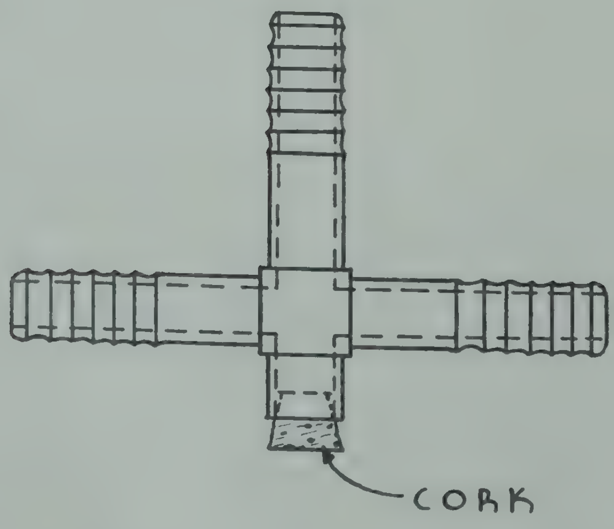


Figure 10B. Fire check for gas line.

thoroughly mixed before entering the burner, as is necessary for both the multiple-tip crossfires and the ribbon burner next described. As a precaution against back-firing of the burners and as a safety measure, a "T" should be inserted in the line, as shown in Figure 10B. When backfiring occurs, the cork is blown from the "T" and no damage is done. Commercial fire checks to prevent this backfiring may be purchased from the Research Vacuum Supply Company.

Multiple-Tip Crossfires

For Pyrex tubing up to 65 to 75 mm. O.D., the burners shown in Figure 11 are satisfactory. The petcock allows easy and accurate adjustment of each burner to produce an identical flame from each. By bending the copper tubing forming the tips, the flame from each burner may be focused so that the hot point of the flame is the desired distance from the burner. For larger size tubing the nine-tip multiple burner, shown in Figure 12, can be

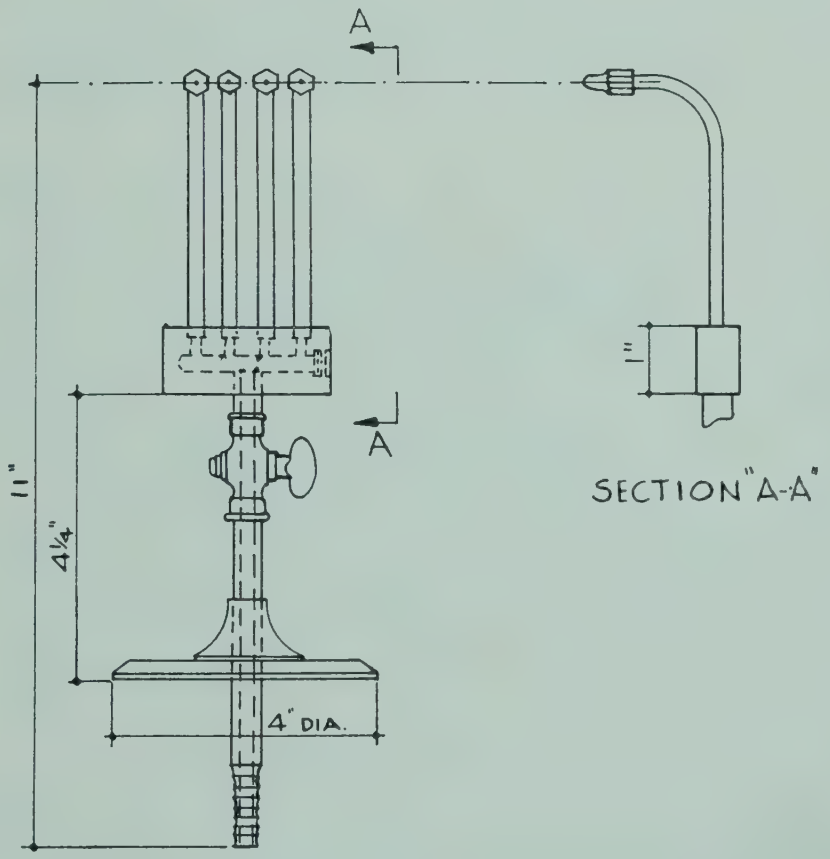


Figure 11. Four-tip crossfires.

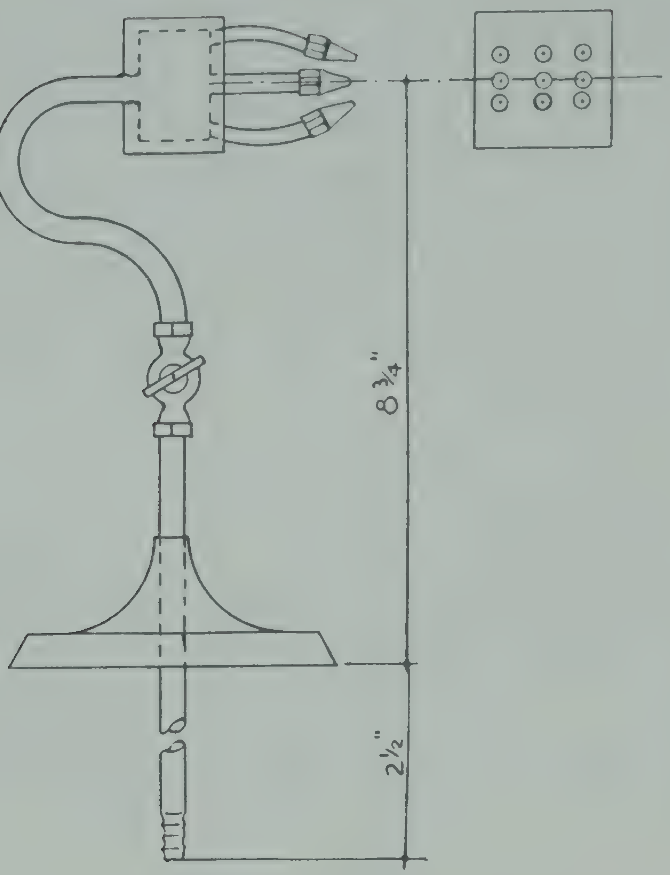


Figure 12. Nine-tip crossfires.

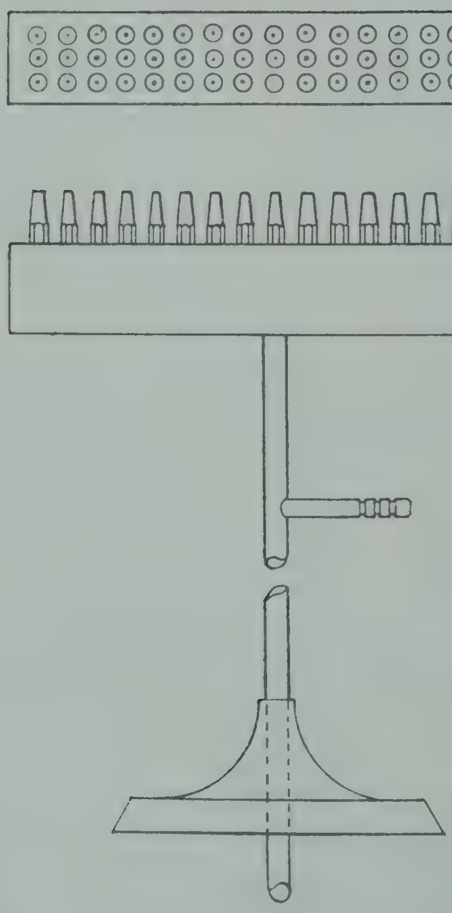


Figure 13. Ribbon burner.

made. These specific burners are not available commercially, but satisfactory substitutes can be obtained.

Ribbon Burner

For heating large sections of tubing, as is necessary when making sweeping right-angle or U-bends, the burner shown in Figure 13 is satisfactory. Most glass coils are wound by heating over this burner. Item No. 19 in Figure 1B, page 25 shows a ribbon burner that can be used for making bends in tubing up to 4 in. in diameter. These burners must be used with the same premixer required for the multiple-tip crossfires.

POWER TOOLS

Many glassblowing shops now have power machines, such as various glass-grinding equipment and glassblowing lathes, as well as electrical equipment, such as annealing ovens, spot welders, and induction furnaces for glass to metal seals.

Glass-Grinding Wheel

Sommer and Macca make several sizes of grinding and polishing wheels. Regardless of the size, a glass grinder may well be considered a basic piece of equipment for a glassblowing shop. With a grinder many operations may be performed, for example bottles and squares may be cut and ground, pieces of flat glass may have the edges ground square or beveled, glass tubing may have the ends ground square with the sides of the tubing or at an angle, as the occasion demands.

Spot Welder

When the glass apparatus to be built incorporates electrical heaters or involves electronic equipment, a spot welder will be needed. Several very excellent commercial spot welders are on the market. The author has used a Litton spot welder, controlled by the timing device from an old x-ray machine. With this timer, the "on" time could be controlled from 1/20 of a second to a full 20 seconds. Thus making possible welds between two very fine wires or two larger pieces of metal. For the widest possible range of work it is important that the spot welder have some type of control for regulating the "on" time of the current.

Annealing Oven

(An annealing oven is the most consistently used piece of equipment in a glassblowing shop. Generally, it is safe to estimate the time necessary to flame anneal a finished piece of glass apparatus as about the same as that spent in construction of the article. When an annealing oven is available, the time spent in flame annealing may be cut in half and in many cases eliminated entirely. For the most efficient service an annealing oven should be so controlled that it will heat to annealing temperature, hold this temperature for a predetermined time, and then shut off automatically. With this type of control the oven may be started at the close of the work day and the glassware will be annealed and cool enough to remove from the oven the following) morning.

In the glassblowing shop at the University of California at Los Angeles, there are two ovens, one a horizontal oven with an inside dimension of 24 × 30 × 48 in., and the other a vertical oven having inside dimensions of 18 × 18 × 72 in. Both of these ovens were made by Lydon Brothers and are automatically controlled. Each oven has its own vacuum system with a high-vacuum manifold running inside, making each suitable for annealing glass or evacuating and degassing glass apparatus. For large ovens such as these, there should be a blower inside to circulate the air and eliminate hot spots. Smaller ovens may be made, and the blower eliminated; a blower, however, is recommended if it is at all possible to install one.

Glassblowing Lathe

With practice most types of apparatus used in chemical research can be made by hand. Even so, the most experienced glassblower will find many uses for a glassblowing lathe.

For laboratories which do not enjoy the services of a fully trained and experienced glassblower, but which do have other personnel showing both interest and natural ability in glassblowing, the acquisition of even a small glass blowing lathe would be a sound investment. (The time saved in blowing a particular piece of equipment would be considerable and, in addition, the quality of the work would be markedly improved.)

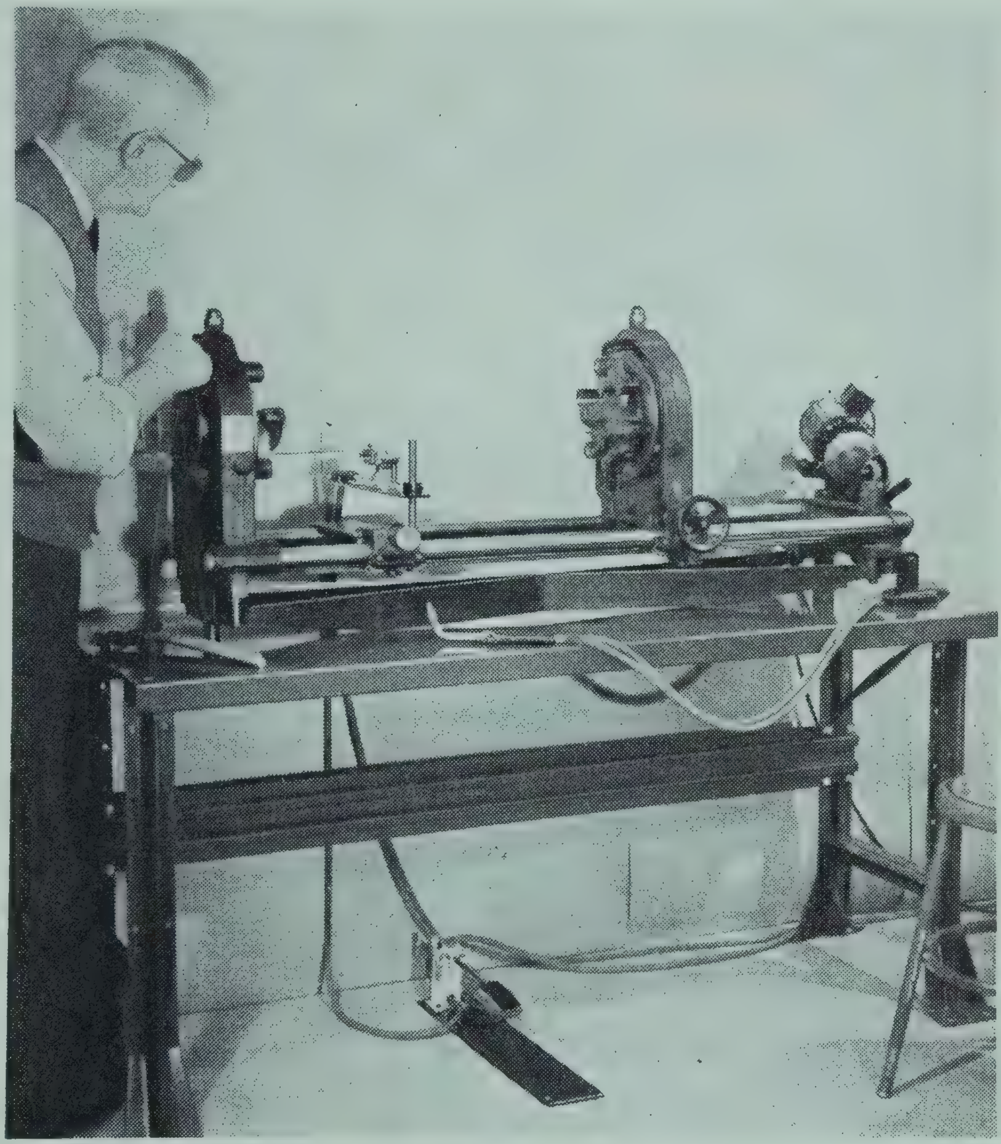


Figure 14. Small glassblowing lathe.

Glassblowing lathes are manufactured by several firms and come in a variety of sizes (see Appendix I for list of manufacturers). Each type of lathe should be investigated to permit the prospective buyer to select the one most suitable to the conditions and problem at hand. A precision glassblowing lathe such as the Litton lathe is in general quite expensive. For highly trained glassworkers this precision is desirable, thus warranting the higher cost. A new glassblowing lathe made by the Bethlehem Apparatus Co., Inc., is now on the market. This lathe was designed to fill the need for a glassblowing lathe in the moderate price range. (see Figure 14).

High-Frequency Induction Heating Unit

High-frequency heating may be used for glass to metal seals, soldering, brazing, annealing of metal, as well as melting and casehardening. An induction heater is also necessary for the successful degassing of Kovar parts preparatory to making glass to metal seals. High-frequency induction heaters are either of the electronic tube generator design or the spark gap design. For commercial production work where continuous duty is of primary importance, the electronic tube generators are probably more satisfactory. The transformers are usually provided with several taps for adjusting the filament and plate voltages. Electronic tube generators require extensive adjustment with each change of work coils—a factor which makes these heaters more suitable for operations which do not require the constant changing of the work coils. The spark gap type of high-frequency induction heating, on the other hand, is particularly adapted to varying types of work. Usually a single knob serves as a tuning regulator and power switch.

Drill Press

A standard floor model drill press with a movable drill table should be obtained. By moving the table up and down, or out of the way, various sizes of work can be accommodated more easily.

Bench Grinder

A bench grinder should be included in the shop equipment as a means of grinding new surfaces on the glass-cutting files, as well as sharpening the various tools which will be used around the glassblowing shop. A floor model grinder can be substituted, although the additional cost will hardly be justified.

Suppliers

Material supplied	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28
Aluminum paint (Heat-resistant)																										×		
Annealing oven (glass)															×													
Asbestos cloth					×			×			×					×							×	×	×			
Asbestos gloves					×			×		×	×												×	×	×			
Asbestos tape					×			×								×							×	×	×			
Carbon rods										×															×			
Carbon paddles										×										×					×			
Diffusion pumps, oil & Hg					×	×				×				×						×				×	×			
Diffusion pump, oil					×					×				×						×				×	×			
Electronic control inst.				×																×								
Furnace muffles																	×			×								
Gas pressure booster												×																
Gas & air burners					×	×	×	×		×	×									×		×	×	×	×			
Gas & oxygen burners	×				×	×	×	×		×	×									×		×	×	×	×			
Glass cutoff wire (hot)										×												×		×	×			
Glass cutoff saw		×								×															×		×	
Glassblower's goggles					×	×		×		×												×	×	×	×			
Glass storage cabinets			×																									
Glass support rollers										×										×		×		×				
Goggles for quartz work								×			×														×			
Grinders (glass)		×								×																	×	
Grinding compounds					×			×									×								×			
Greases (high-vacuum)				×		×		×		×													×	×	×			
Hand tools (glassworking)					×	×		×		×												×		×	×			
Induction furnace													×															
Ionization gage																												
Kovar-glass seals																												×
Lathes (glassworking)			×							×				×								×						
Leak detectors (vacuum)									×																			
Marking pencils (glass)					×			×		×										×		×	×		×			
Marking ink (glass)					×			×		×										×		×	×		×			
Oxygen regulators	×				×			×		×												×	×					
Polariscopes										×									×			×						
Polishing equipment (glass)																		×										
Pirani gages										×																		
Spot welder										×			×								×	×						
Tesla high-frequency coil			×		×	×		×		×												×	×	×	×			
Tungsten kulgrid wire leads		×	×		×	×		×		×												×	×	×	×			
Vacuum pumps		×	×		×	×		×		×		×		×								×	×	×	×			

List of Suppliers and Addresses

1. Air Reduction Co.
   New York 17, N.Y.
2. American Instrument Co.
   Silver Springs, Md.
3. Bethlehem Apparatus Co.
   Hellertown, Pa.
4. Biddle Co. James G.
   Philadelphia 7, Pa.
5. Braun Corporation
   Los Angeles, Calif.
6. Callite Tungsten Corp.
   Union City, N.J.
7. Carlisle Gas Burner Equipment
   Millville, N.J.
8. Central Scientific Co.
   Chicago, Ill.
9. Consolidated Vacuum Corp.
   Rochester 3, N.Y.
10. Eisler Co., Chas.
    Newark, N.J.
11. Gordon Duff & Co.
    West Los Angeles, Calif.
12. Leiman Brothers Inc.
    Newark, N.J.
13. Lepel High Frequency Lab.
    Woodside 77, N.Y.
14. Litton Industries
    Grass Valley, Calif.
15. Lydon Brothers
    Hackensack, N.J.
16. Manville, Johns
    Los Angeles, Calif.
17. Norton Co.
    Worcester, Mass.
18. Optical Equipment Co.
    Los Angeles, Calif.
19. Polaroid Corp.
    Cambridge, Mass.
20. Precision Distillation Apparatus Co. Santa Monica, Calif.
21. Raytheon Mfg. Co.
    Waltham, Mass.
22. Research Vacuum Supply Co.
    Chicago, Ill.
23. Sargent, E. H. & Co.
    Chicago, Ill.
24. Schaar & Co.
    Chicago, Ill.
25. Scientific Glass Apparatus
    Bloomfield, N.J.
26. Sheffield Bronze Paint Co.
    Cleveland 19, Ohio
27. Sommer & Maca
    Chicago, Ill.
28. Stupakoff Ceramic Mfg. Co.
    Latrobe, Pa.
29. Western Scientific Co.
    Berkeley, Calif.

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IV

BASIC OPERATIONS

IDENTIFICATION OF DIFFERENT GLASSES

Glasses with different chemical compositions have different expansion coefficients and should be stored in entirely separate cabinets and carefully labeled. When there is a question as to the identity of a piece of glass, some form of identification test should be made. Sometimes this identification can be made by eye: soft glass, for example, often has a heavier blue cast than Pyrex tubing. Visual inspection is not, however, especially reliable. A better method is to make comparison with a known piece of glass. This can be done in several ways, described as follows.

Soft Glass

Using a gas-air flame, an attempt can be made to seal a piece of soft glass tubing to the glass being identified. If the unknown glass is also soft glass, a satisfactory seal will be made quickly and smoothly. If the glass being tested is Pyrex glass, it will not soften enough to be worked in the gas-air flame.

Pyrex Glass

A second method of testing is to seal a small piece of the unknown glass on one end of a piece of Pyrex glass rod, working the unknown glass into a ball by rotating in a gas-oxygen flame.

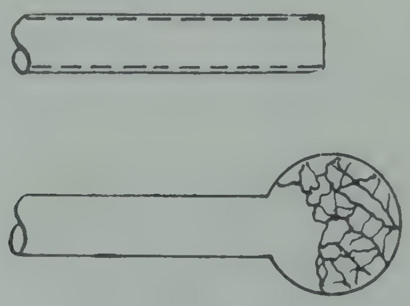

Figure 1. Testing composition of unknown glass.

The ball can then be cooled quickly in a stream of air, and if the two glasses do not have a similar composition, the glass union will show a series of checks and cracks, as illustrated in Figure 1.

Drawing of Two Rods

A comparison of the coefficient of expansion of two pieces of glass can be obtained by forming a rod of the glass being tested and holding it beside a similar piece of glass rod whose composition is known, as shown in Figure 2A. The ends of the two pieces of rod should then be heated and pressed together to assure a good contact, as illustrated in Figure 2B. After the two ends are heated evenly again, they should be gripped with a pair of tweezers and, while the glass is still hot, pulled to a small fiber of about ½ mm. O.D.; the glass fiber should then be allowed to cool while still being held taut. If on being released the drawn-out section remains straight, the two glasses have approximately the same expansion. If it curves, the glass on the inside of the curve has the higher coefficient of expansion. See Figure 2C.

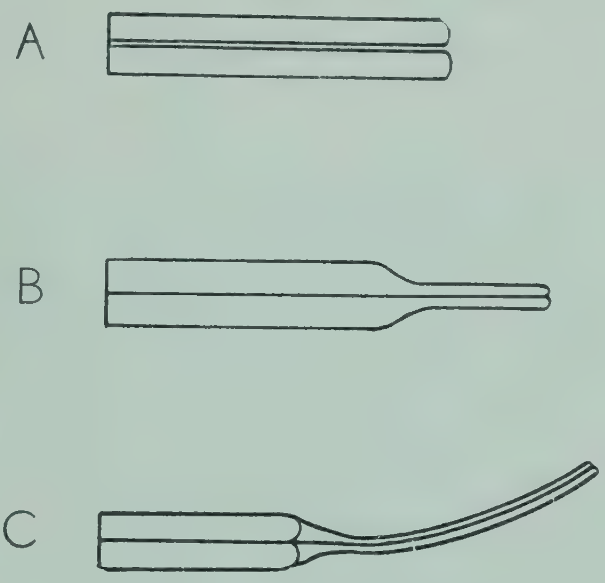

Figure 2. Identification of glass by drawing.

Immersion in Liquid

If a number of pieces of soft glass and Pyrex glass tubing have become mixed, a quick but accurate check of these can be made using a solution of 59 volumes of carbon tetrachloride and 41 volumes of benzene. This forms a solution with a refractive index approximately that of Pyrex glass. When the assortment of glass tubes is placed in the solution, the soft glass tubing will be clearly visible, whereas the Pyrex glass will become invisible.

CLEANING OF GLASS TUBING

Glass should be thoroughly cleaned before any glassblowing is attempted, since dust or foreign material may cause the glass to devitrify while it is being heated. In extreme cases when the glass has not been properly cleaned, the apparatus may crack after it is completed and cooled, even when it has been properly annealed. Merely running water through the tubing is not sufficient, as the water may not remove all the particles of dirt. A wet rag should be forced through the tubing by means of a wooden stick. It is good practice to avoid metal or glass rods for this operation, since they may scratch the walls of the tube being cleaned. The outside of the tube should also be rinsed with water and wiped with a damp cloth, and both the inside and outside wiped with a clean, dry cloth.

Care should be taken to keep finger marks from the glass, since these marks readily burn into the glass and once there, are very hard to remove. Cleanliness of the hands is very important.

Cleaning Apparatus To Be Repaired

Apparatus which is to be repaired should be cleaned as thoroughly as new tubing. Quite often this is not as easy as with new tubing, but immersion in an acid or solvent bath or the use of soap and water will produce the desired results.

Caution: All traces of vapors from any solvents must be removed before starting work, to avoid the hazard of an explosive mixture of solvent and air.

CUTTING GLASS TUBING

Cutting Glass Tubing with a File

For cutting glass the most widely used method is with a file, and a flat machinist's file is satisfactory. These files can be resharpened when the edges become dull. In making a cut with the file, the glass should be scratched about one-third of the way around the circumference of the tube at the point where the break is desired. An inexperienced worker will find it advantageous to practice with glass tubing of about 6 to 8 mm. O.D. In time, however, he will find it possible to break glass tubing up to 1½ in. in diameter this way. It is better to make the scratch-mark with one stroke of the file, using considerable pressure. The file is held perpendicular to the axis of the tubing and pushed forward, while at the same time the tubing is rotated with the top moving toward the worker. See Figure 3. For larger tubing a second scratch may be used, starting at the edge of the first mark and extending further around the circumference of the tube. The purpose of making the scratch is to fracture the surface and create a strain at the desired point. It is not advisable to use the file as a saw, since a sawing motion merely tends to widen the scratch-mark, making the tube less likely to break even, and bends the teeth of the file, making it dull for future scratches. After the

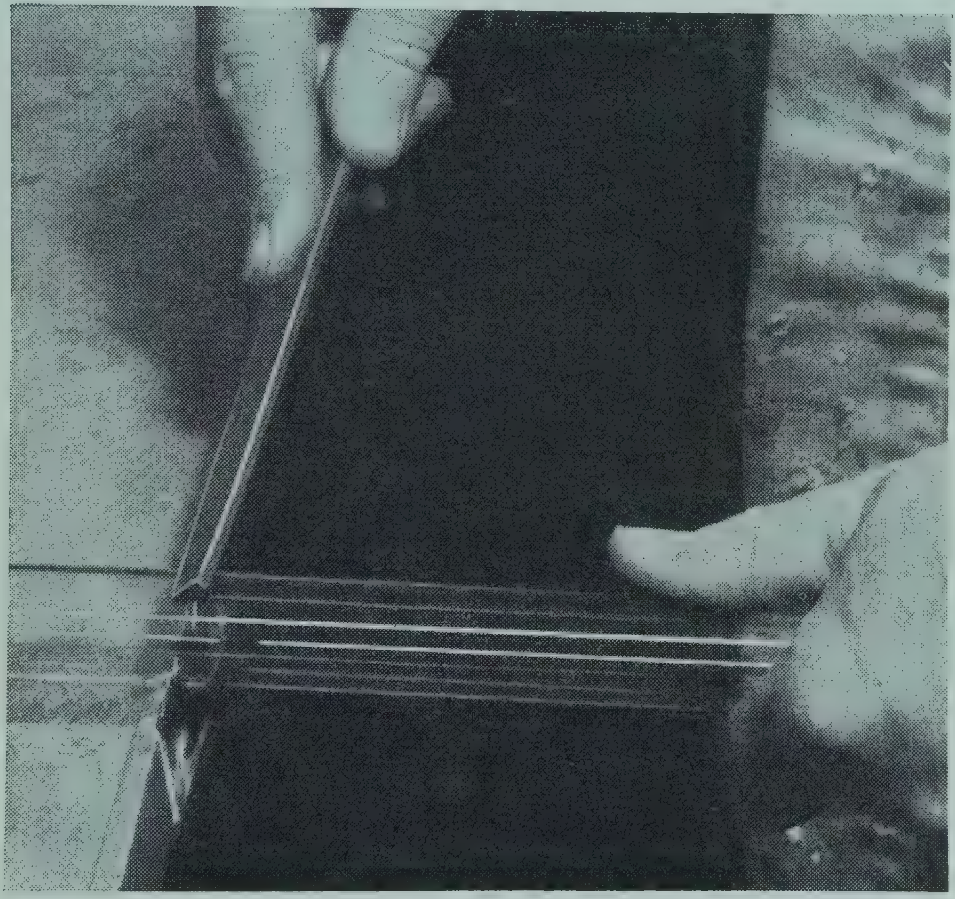

Figure 3. Making scratch mark on glass tubing.

scratch-mark is completed, the tubing is laid across the cutting edge in position to be gripped firmly by the right hand which is resting on the work bench. A steady pressure down and away from the bench is maintained, on the other end of the glass tube, by the left hand. See Figure 4. The result will be a smooth fracture. This break will be made more easily if a drop of water is placed on the scratch-mark before attempting to complete the break. Milligan[1] has found that the strength of glass containing cracks is greatly influenced by a liquid in contact with the surface of the glass. Exposure of the surface to air saturated with alcohol or water greatly reduces the strength. Putting a drop of water on the crack reduces the strength by about 20%, where as moistening the crack with dry paraffin oil strengthens the tube by about 20 %. Milligan does not advance an explanation for either of these effects, but many glassblowers can attest to the accuracy of the statement that water in the scratch reduces the strength of the glass tubing.

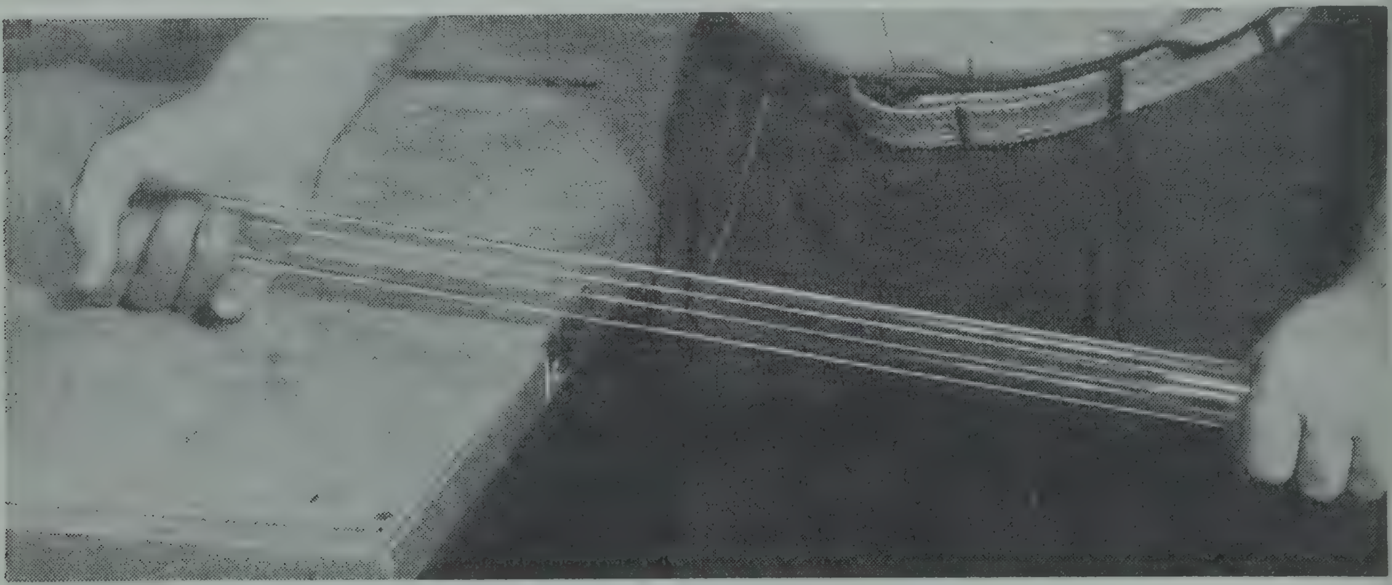

Figure 4. Breaking glass with pressure

Cutting Small Tubing with a File

The tube may be broken by making the usual file scratch, after which it is gripped firmly in the hands. The scratch-mark is between and opposite the thumbs, as shown in Figure 5. The tube is then snapped with a combined pulling and binding operation. If a small piece of glass breaks from the edge of either or both pieces of tubing during the breaking process, it means the glass was bent too sharply and should have been pulled more.

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[1] Milligan, L. H., *J. Soc. Glass Technol.*, **13**, 351 (1929).

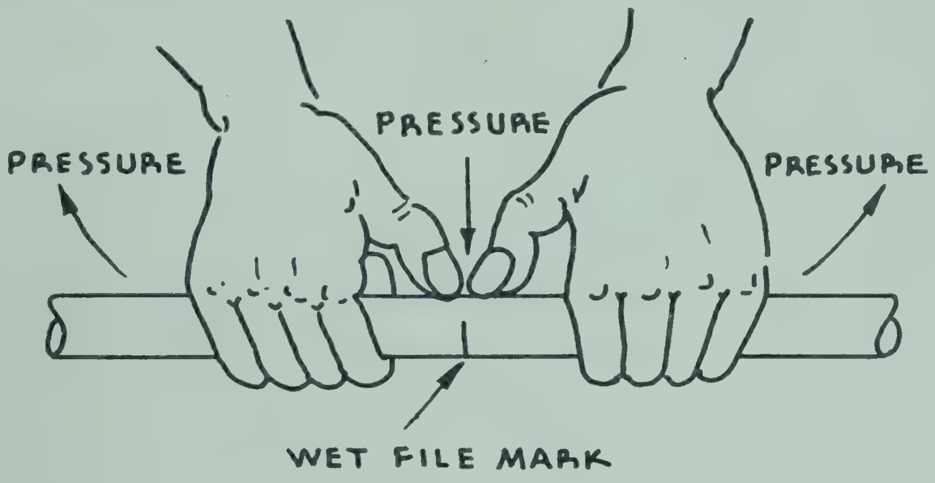

Figure 5. Breaking small tubing in hands.

After several cuts the glassworker should know approximately the amount of stress needed to produce a straight fracture. If considerably more stress is required, the break will not be smooth and the scratch-mark should be deepened by a second application of the file.

Cutting Short Pieces with a File

Short pieces of small glass tubing, or pieces too hot to hold in

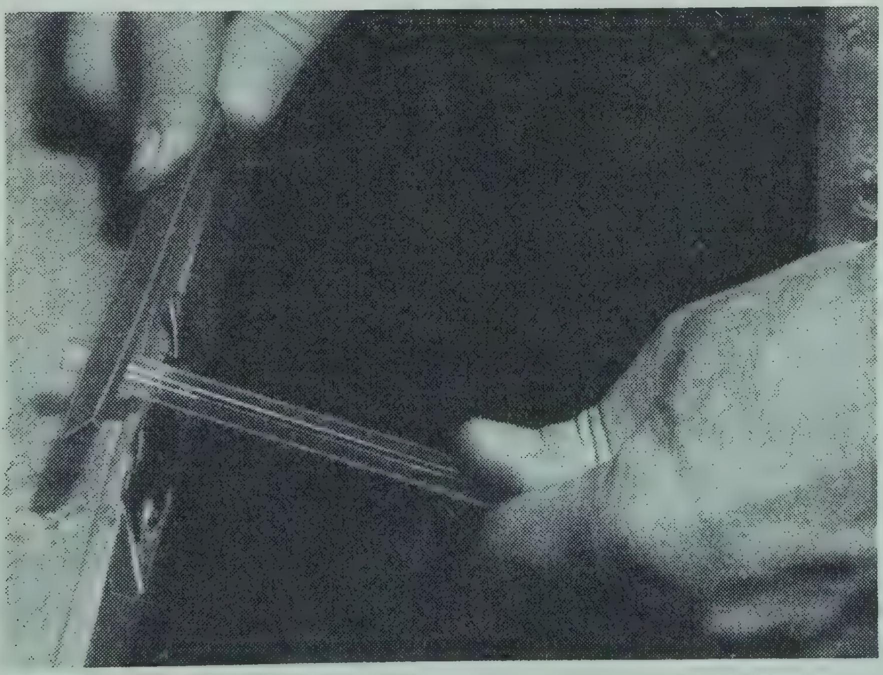

Figure 6. Cutting short pieces of glass

the hands, may be broken by making the file scratch, then placing the scratch on the slotted cutting edge, and striking the end of the tube a glancing blow with the file, as shown in Figure 6. For larger tubing (above 22 mm. O.D.), better results may be obtained by immersing the file in water before making the scratch-mark, and then striking it a glancing blow with the file as with smaller diameter tubing.

Cutting Glass with a Hot Rod

Large-diameter tubing, that cannot be cracked by striking with a file, requires a different technique. After being scratch-marked with a file, the tubing may be cracked by applying the tip of a small piece of Pyrex rod (heated white-hot in an oxygen-gas flame) to one end of the file mark. The crack started this way may not go all the way around the circumference of the glass tube. If not, it can be made to do so by leading the crack with repeated applications of the hot glass rod, each application being just in front of the crack. This method is especially convenient on completed and assembled apparatus such as vacuum systems. See Figure 7.

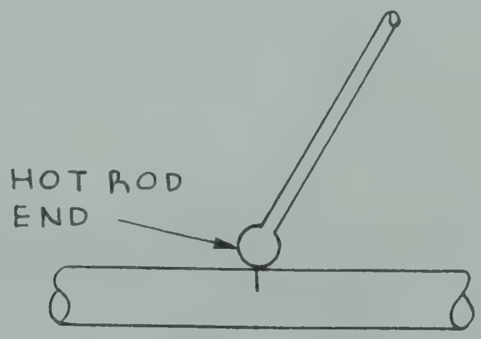

Figure 7. Cutting glass with hot rod.

Hot Wire Cutting of Pyrex

For cutting larger sizes of Pyrex brand tubing as well as bottles, the electrically heated wire cutter shown in Figure 8A is very useful. The glass is laid in the V-track support rack and the wire wrapped around the tube at the spot where the break is desired. The wire should be held taut by pulling on the handle while the current is turned on by depressing the foot pedal to the halfway mark. Care should be taken to see that the loop is not

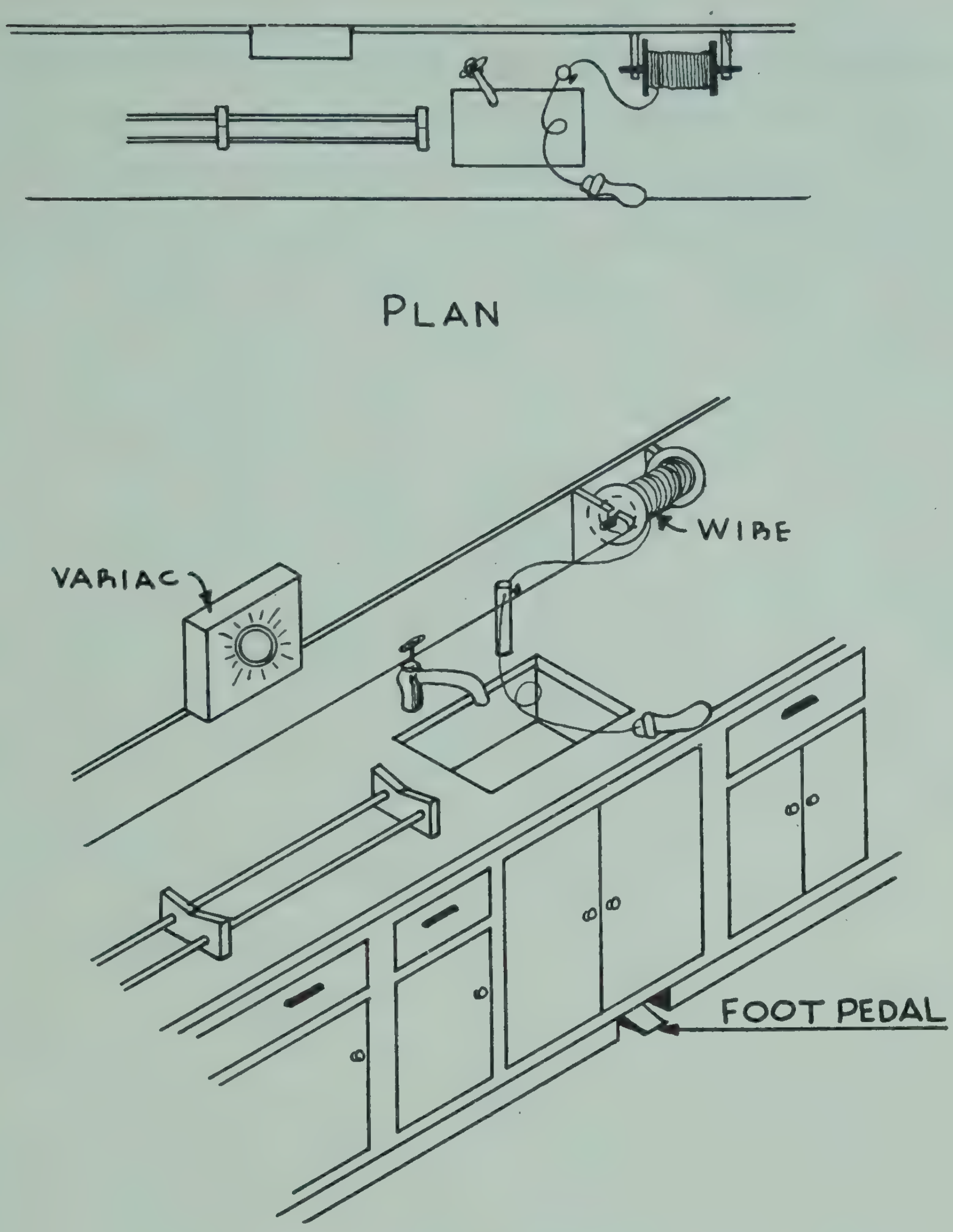
PLAN
VARIAC
WIRE
FOOT PEDAL

Figure 8A. Hot wire cutter.

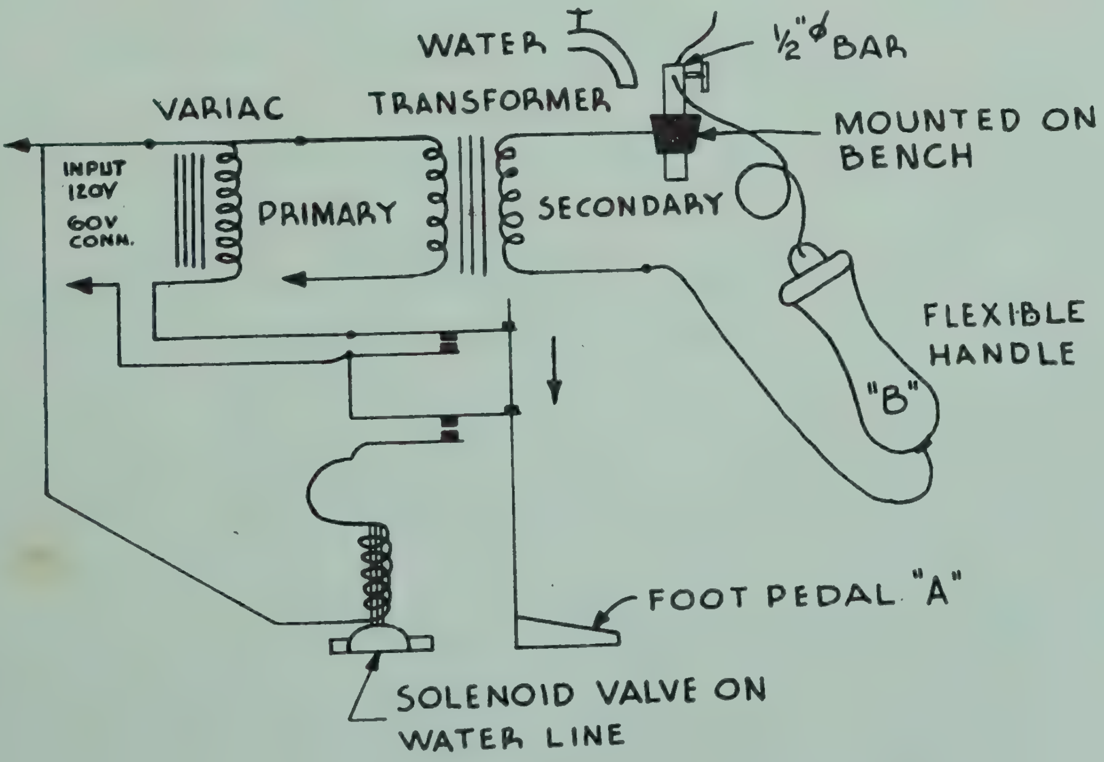
WATER
½"ϕ BAR
VARIAC
TRANSFORMER
MOUNTED ON BENCH
INPUT 120V 60V CONN.
PRIMARY
SECONDARY
FLEXIBLE HANDLE
"B"
FOOT PEDAL "A"
SOLENOID VALVE ON WATER LINE

Figure 8B. Circuit for hot wire cutter.

short-circuited at any point. After a few seconds of red heat in the wire, the foot pedal should be completely depressed, thus turning on the cold water, which is controlled by a solenoid valve. If the glass tube does not break with this treatment, it is an indication that a higher wire temperature is required. In this case the Variac should be adjusted higher and the operation repeated. The hot-wire method of cutting glass will produce a clean crack completely around the circumference of the tube.

The voltage to the resistance wire should be so regulated that the wire comes to a red heat completely around the periphery of the glass tube as quickly as possible. When heating is too slow, the surface of the glass is heated over a wider area and cracks running longitudinally down the tube will result. Small irregularities in the cracked edge may be removed by grinding or by peeling, or, when not too pronounced, they may be smoothed over by heating the end of the tubing and shaping it with a carbon paddle.

Hot Wire Cutting of Soft Glass

The procedure is very similar to that used for cutting Pyrex glass, with the exception that water is not used. The wire is brought to a red heat and held tightly around the glass for a few seconds, after which it is quickly released from around the glass. The shock of the cool air will usually result in the glass breaking. If the glass does not break at once, the tip of the finger should be moistened and placed on the glass at the point where it was encircled by the hot wire. If this is done quickly while the glass is still hot, the crack will run completely around the glass tube. To ensure a smooth cut at right angles to the axis of a bottle, it is advisable to circle the bottle with a diamond scratch before using the hot wire. The resulting cut will be smoother, thus reducing the grinding time necessary to obtain a flat edge on the bottle.

Cutting Bottles with a Flame

At times it may be necessary to cut soft glass containers which are too large to fit in the hot wire assembly. These may be cut by means of the turntable shown in Figure 9. Small wooden blocks are fastened in position at the base of the bottle to hold it centered on the turntable, which is then rotated as fast as possible without disturbing the balance of the bottle. A hand torch is clamped in position as shown, with hot flame directed at the side of the bottle. After a number of revolutions the glass vessel should crack cleanly and squarely. This end should then be ground flat on the grinding wheel, or it may be done on a piece of flat plate glass, using Carborundum and water. The glass should be allowed to cool before grinding is attempted. Soft glass vessels cannot be fire-polished and therefore must be finished off by grinding.

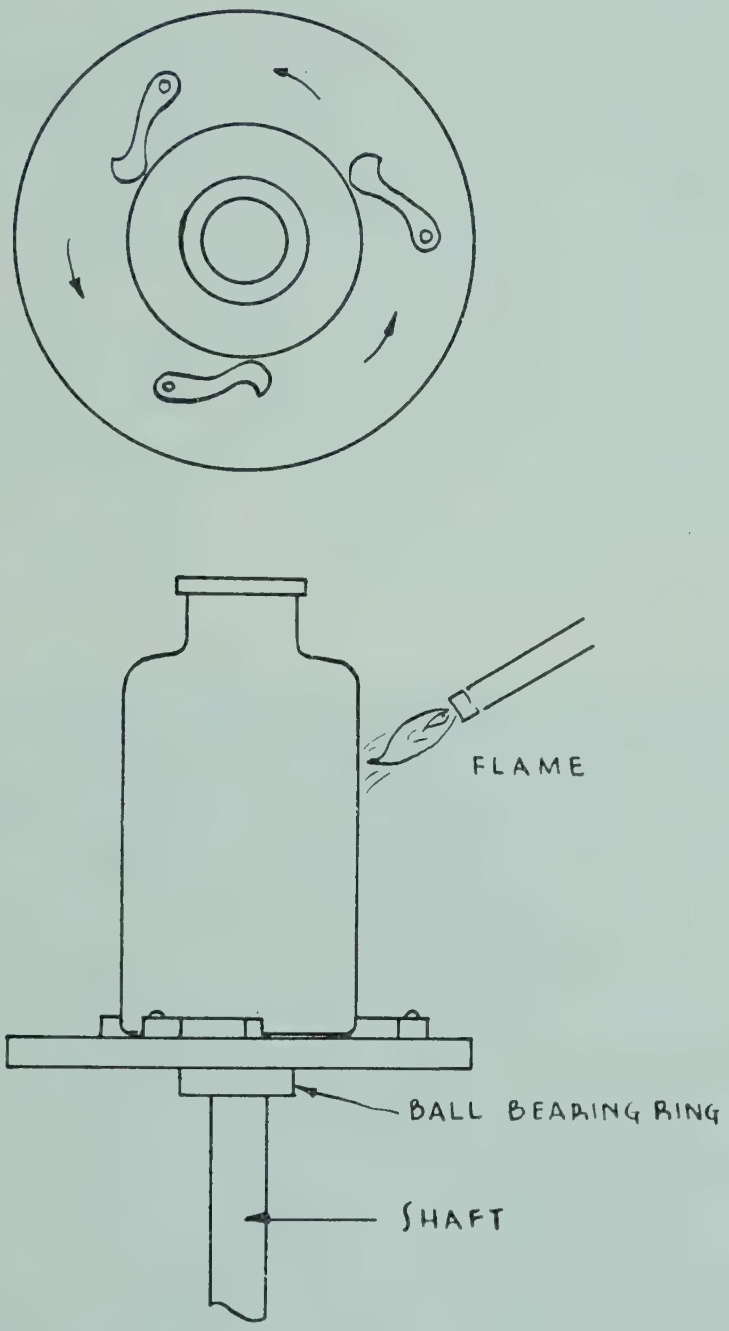

Figure 9. Cutting a bottle by means of a flame.

An edge which while rough will not be a cutting edge may be obtained by filing the edges, first on the outside and then on the inside of the bottle.

Glass-Cutting Saw

A carborundum cutting-saw is a useful piece of equipment in a glassblowing shop. A glasscutter made by the American Instrument Company has 12-in. bonded abrasive wheels and can be used to cut any desired angle. Glass cut on this saw should be thoroughly washed before being used.

The sawed surface of the glass is semipolished and leaves a slight gray line when sealed to another piece of glass. This line can be eliminated by fire-polishing the sawed edge before attempting the seal.

Fire Cutoff

A method most commonly used by glassblowers, and not always mentioned, is the fire cutoff. In this the tubing is sealed off as in making test tube ends. For small tubing the whole end

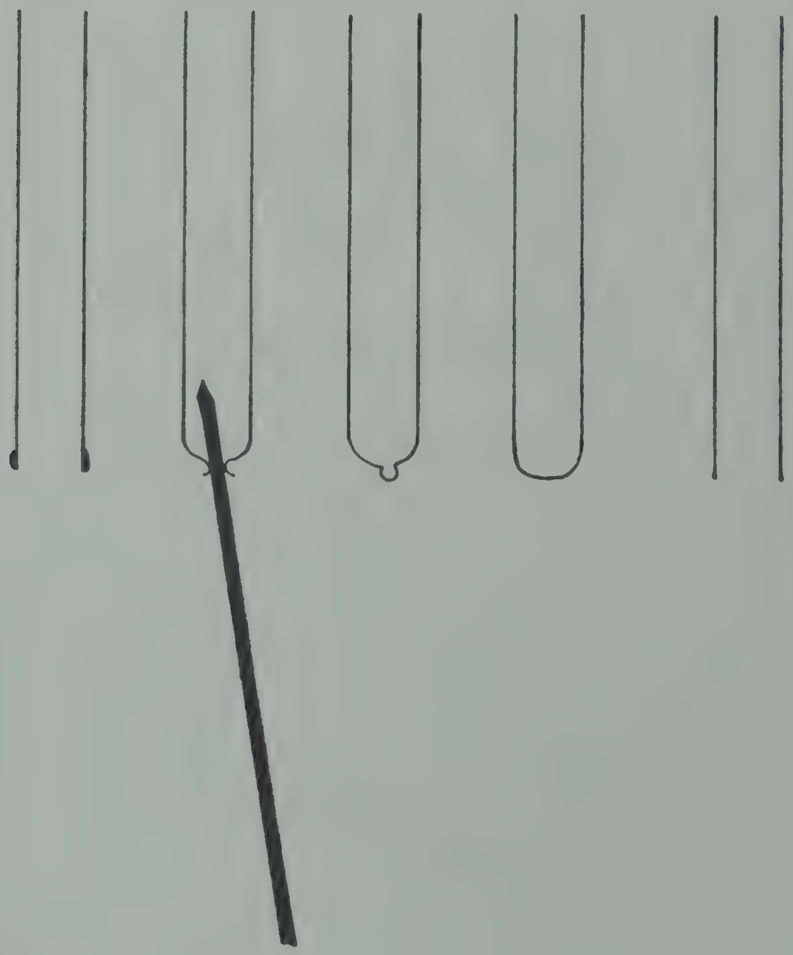

Figure 10. Fire cutoff.

should be reheated and blown out. For larger glass tubing a small section of the end should be blown out and this hole flared with a carbon rod as in Figure 10. Either of these methods gives clean fresh surfaces that can be sealed to another piece of glass without leaving any visible mark.

Cutting Glass with Scissors

Pieces of Pyrex tubing can be cut, shortened, or modified by bringing the glass to a red heat with a gas-oxygen flame and then cutting with a pair of scissors. The glass can be cut only while at a temperature high enough to make it soft or pliable.

Rough shapes such as squares, circles, and triangles may be cut from window glass with a pair of scissors, provided the cutting is done under water. For this purpose a large pair of tailor's scissors and a pail of water are adequate.

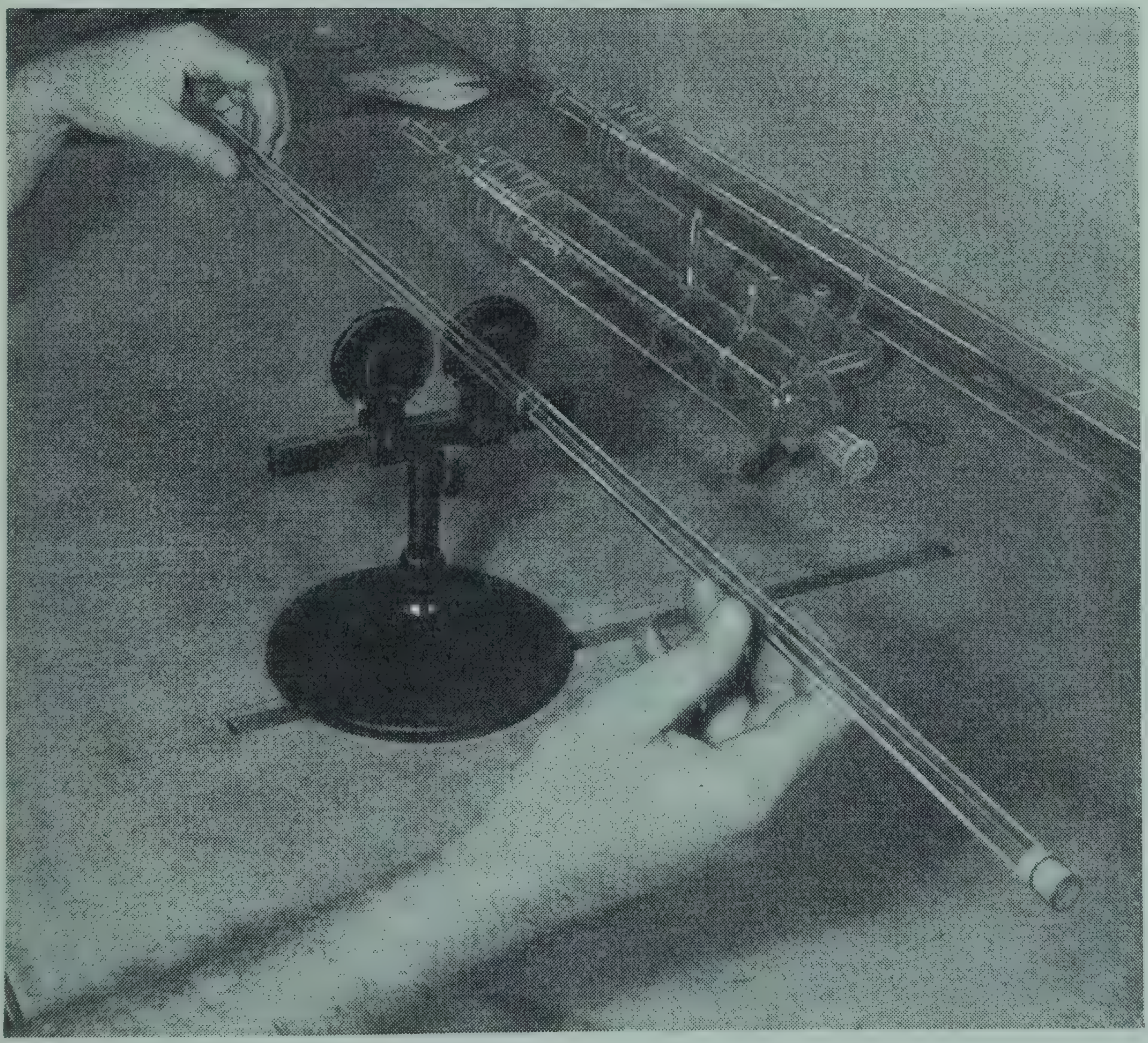

Figure 11A. Position of hands for rotating the glass.

ROTATION OF GLASS TUBING

Uniform rotation of the glass during the heating and blowing operation is essential for obtaining even wall distributions and symmetrical shapes. The tubing is usually held by the little finger of the left hand, with the tubing end nearest to the burner resting on a stand or roller as shown in Figure 11A. The thumb and forefinger are used to rotate the glass tubing. The right hand supports the other piece of tubing to be sealed. Here again the thumb and forefinger are used to rotate the tube while the other fingers are used mainly for support. Note that the palm of the left hand is down and the palm of the right hand is up. These positions permit the glassblower to drop into the second position easily (Figure 11B). This second position is used for bringing the axis of the finished seal into line. Rotation may be in either

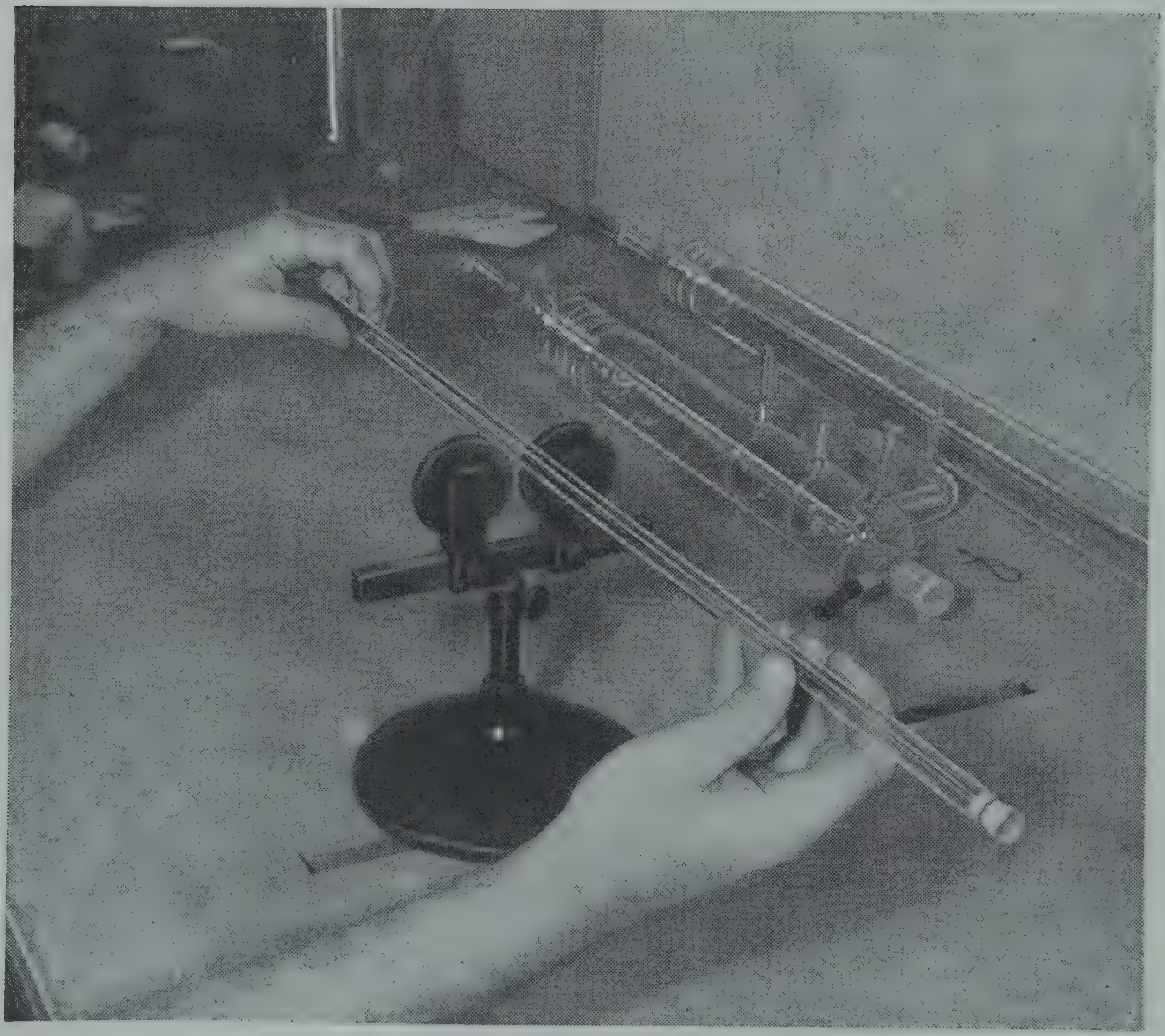

Figure 11B. Position of hands for trueing glass during rotation.

direction, or back and forth; however, the better method is to rotate in such a way that the top of the tube moves away from the glassblower.

Points to Remember in Rotating

1. The tube should not be pushed or pulled unless it is necessary to constrict or change the wall thickness or the diameters of the glass tubing.

2. The speed of rotation of the right and left hand should be synchronized to prevent twisting the tubing.

3. Both tubes should be held in a straight line; they should not be allowed to bend or sag at the heated zone.

Practice Pieces for Rotating

A good practice piece for rotating may be made as shown in Figure 12.

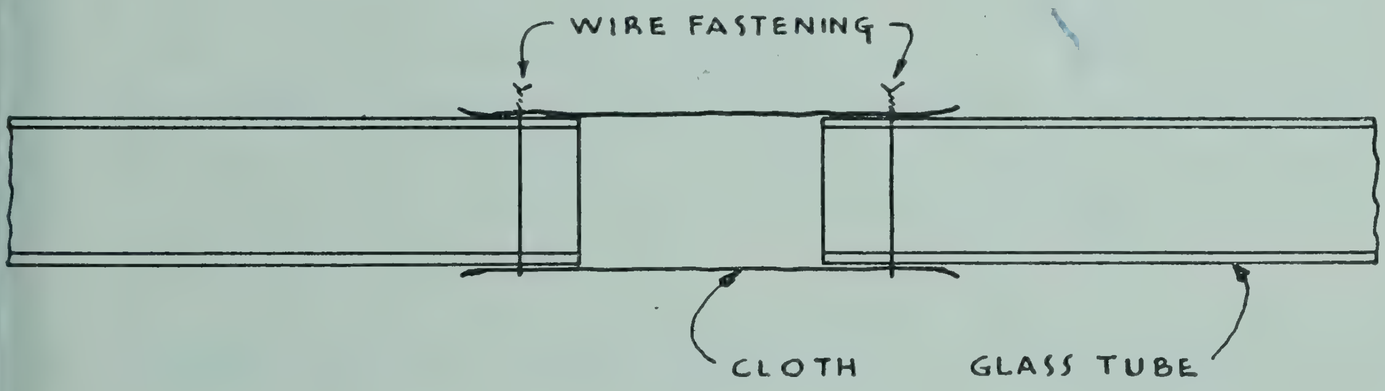

Figure 12. Practice piece for rotating.

Diameter and Wall Thickness Guide

1. Pulling the glass decreases the diameter and wall thickness.

2. Blowing the glass increases the diameter and decreases the wall thickness.

3. Heating the glass decreases the diameter and increases the wall thickness.

4. Pushing an enlarged tube increases the diameter and the wall thickness.

Choice of Glass for Practicing

The choice of glass used will depend on the facilities available. Soft glass can be worked in a gas-air flame, eliminating the expense of oxygen. Soft glass is also less expensive than Pyrex. Both soft glass and Pyrex glass require about the same technique for working, but soft glass, because of its higher expansioncoefficient, tends to crack more easily. For this reason some glassblowers prefer teaching with soft glass because it develops better work habits than when Pyrex is used. With soft glass, except for the most simple operation, the various sections must be kept hot or cracking will result. Because of the need to minimize this danger, the practices of preheating, keeping the work hot, and carefully annealing the finished work become automatic.

With Pyrex glass, because of its lower coefficient of expansion and greater resistance to thermal shock, the danger of cracking is greatly reduced. This sometimes gives the inexperienced worker a false sense of confidence which often leads to faulty work habits. A person who has learned to work with soft glass finds the transition to Pyrex easier than the reverse. The use of blue glass for beginners has been recommended because of the visual color changes, although it is doubtful if this advantage is worth the additional cost.

PRELIMINARY GLASSBLOWING OPERATIONS

Fire-Polishing and Flared Ends

It is the usual practice in glassblowing to fire-polish the ends of all tubes. This removes the sharp edge left by cutting and, in addition, makes the edge mechanically stronger.

Flaring is accomplished by heating the edge of the tube until it has shrunk about 2 to 3 mm. The tube is removed from the flame and rotated against a carbon rod held in the opposite hand as shown in Figure 13A. This will cause the edge of the tube to turn out to a diameter 2 to 3 mm. larger than the rest of the tube.

Flares by Spinning

For making large flares, or flares which are spun out at right angles to the axis of the tubing, the tube should be cut on a glass-cutting saw if possible, or ground flat. If facilities for either of these methods are not available the glass tube should be cut and the edges trued by heating and paddling it with a carbon paddle. The tube to be flared should be supported on bench-roller-stands, with the stoppered end farthest from the flame. A fast rotating spin may be obtained by moving the palm of the hand back and forth across the tube. In making the flares it is not necessary that the rotation be all in the same direction. It is important, however, that a fast spin be produced. Spinning alone is sufficient to put a medium flare on a tube, but the flaring action can be hurried, or a larger flare made, by using a carbon rod as shown in Figure 13B. A carbon paddle should be used to bring the flare to right angles and for the final trueing up.

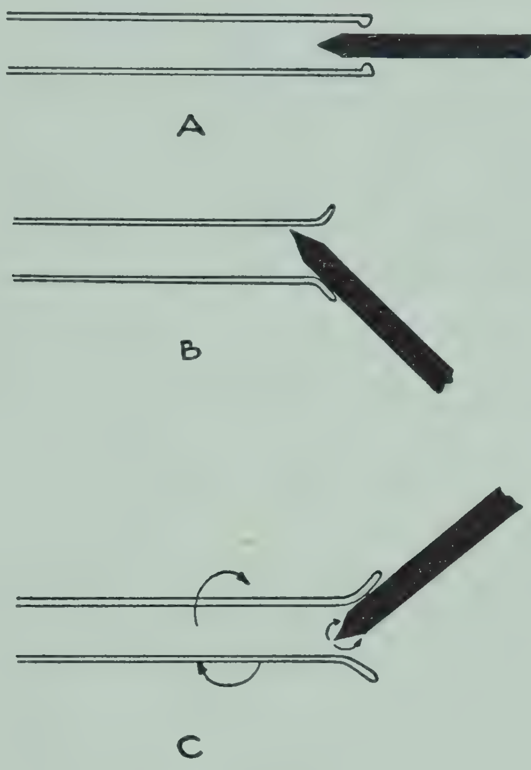

Figure 13. Making flares by spinning the glass.

Flaring Short Pieces of Tubing

Short pieces of glass tubing which are not easily adaptable to the fast spinning method may be flared by resting the end of the tubing on a roller and making the flare in the flame by means of a carbon rod. The glass is rotated in such a way that the top of the tube moves away from the glassblower, while at the same time the carbon rod (6 to 10 mm. O.D.) is swung in an arc with the direction of motion opposite to that of the glass. Moving the glass and the carbon rod in opposite directions tends to smooth out irregularities in the flare which would be accentuated if both moved in the same direction (Figure 13C).

Flares in Stationary Glass Tubing

Flares in stationary pieces of glass tubing or glass which can not be rotated should be made by means of the hexagonal carbon rods described on page 34. When these carbon shapers are to be used, the end of the glass tube should be heated by moving the flame of a hand torch back and forth until the glass is heated evenly. When the glass begins to thicken, but before it shrinks out of shape, the carbon rod should be inserted and quickly rotated back and forth a few times or until the glass sets.

Points

A handle for holding short pieces of tubing may be made by heating the tube in the middle and pulling it as shown in Figure 14. This is done by rotating the tube in a flame until a ½ in. length is heated. When the glass is pliable it should be removed from the flame, and while still being rotated, pulled slowly about 8 to 10 in. This drawn-out section may be melted apart at the center, thus closing both "points." If the points are not in line with the rest of the tubing, it will be necessary to heat them at the juncture with the large tube and straighten them.

Making points is excellent practice in rotating and points may be used as handles and for attaching the blow tube for blowing into the work.

Handles from Glass Rods

A handle can be made from a glass rod by heating the end of the tubing, attaching the rod, and while still rotating, drawing off the soft glass until the end closes up. A fresh end of glass rod should then be sealed to the tip of the pulled-off section, taking care to see that the axis of the rod lines up with the axis of the tubing. The open end of the tube should be fitted with a rubber

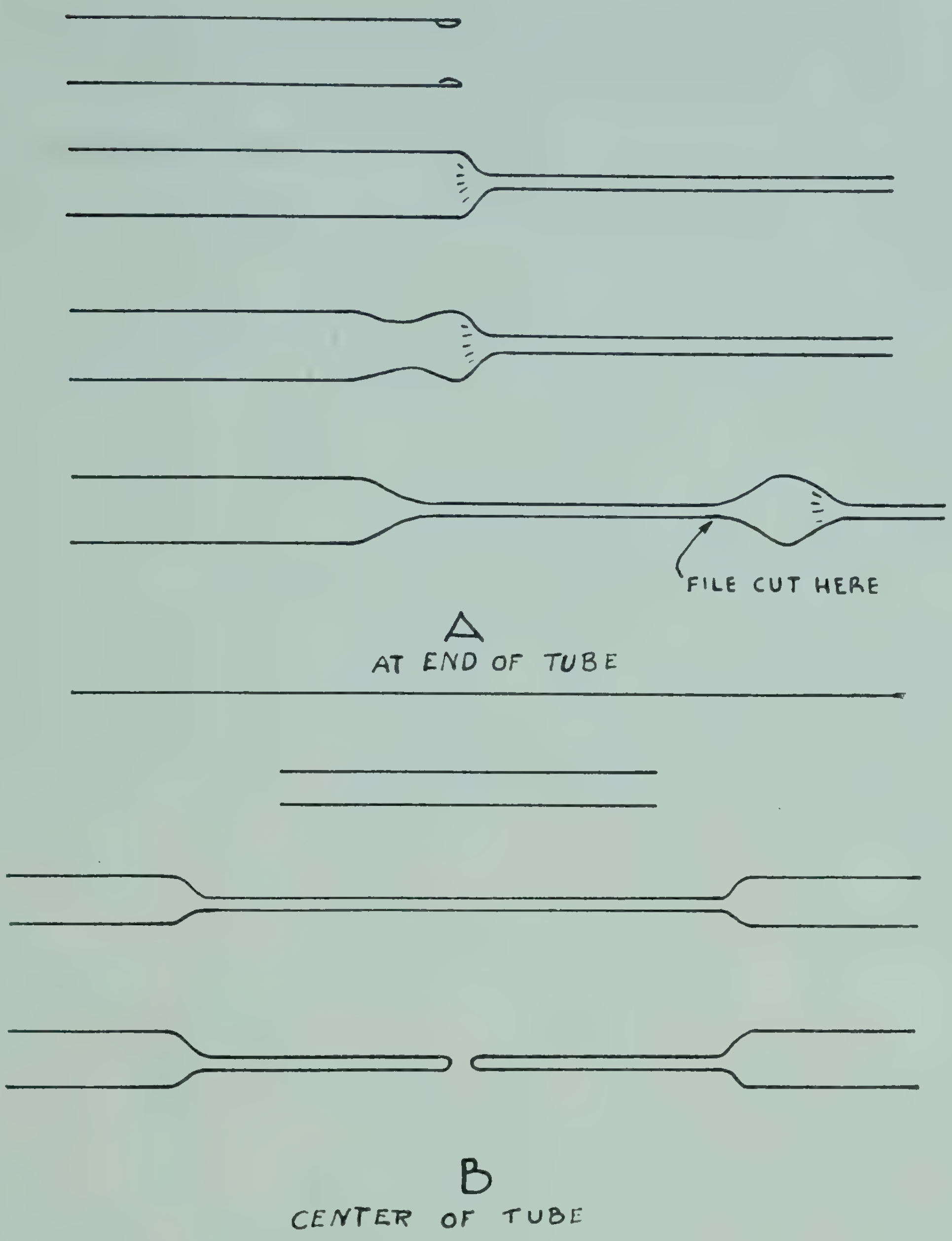
FILE CUT HERE
A
AT END OF TUBE
B
CENTER OF TUBE

Figure 14. Points in glass tubing.

stopper for attaching a blow tube and blowing while the glass is in the flame. The advantage of this over heating the glass, placing it to the mouth, and blowing will be readily apparent if the beginner tries both methods.

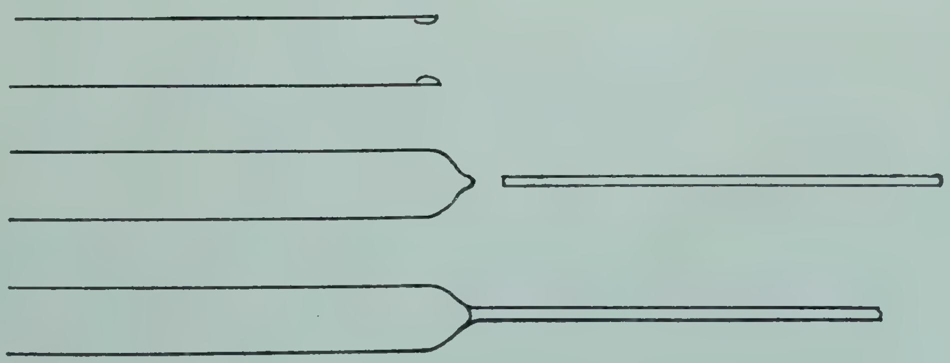

Figure 15. Handles from glass rods.

Round-Bottomed Tubes

A round-bottomed tube is formed by the same procedure used for attaching a glass rod handle, except that, after the tube is closed off, the end is heated uniformly in the flame to allow even distribution of the glass over the rounded surface. See Figure 16. The heavy drop or globule of glass that remains at the tip of the sphere may be removed by touching a piece of rod to the softened glass and pulling off the excess glass. Tweezers may also be used for pulling off some of the excess glass. The closed end should be reheated until the distribution of glass appears to be uniform, at which time it should be removed from the flame and blown into a spherical shape. Several puffs of air should be sufficient and the tube should be rotated continually until the glass sets. If the tube is held vertical for the final blowing, the round bottom should be more symmetrical.

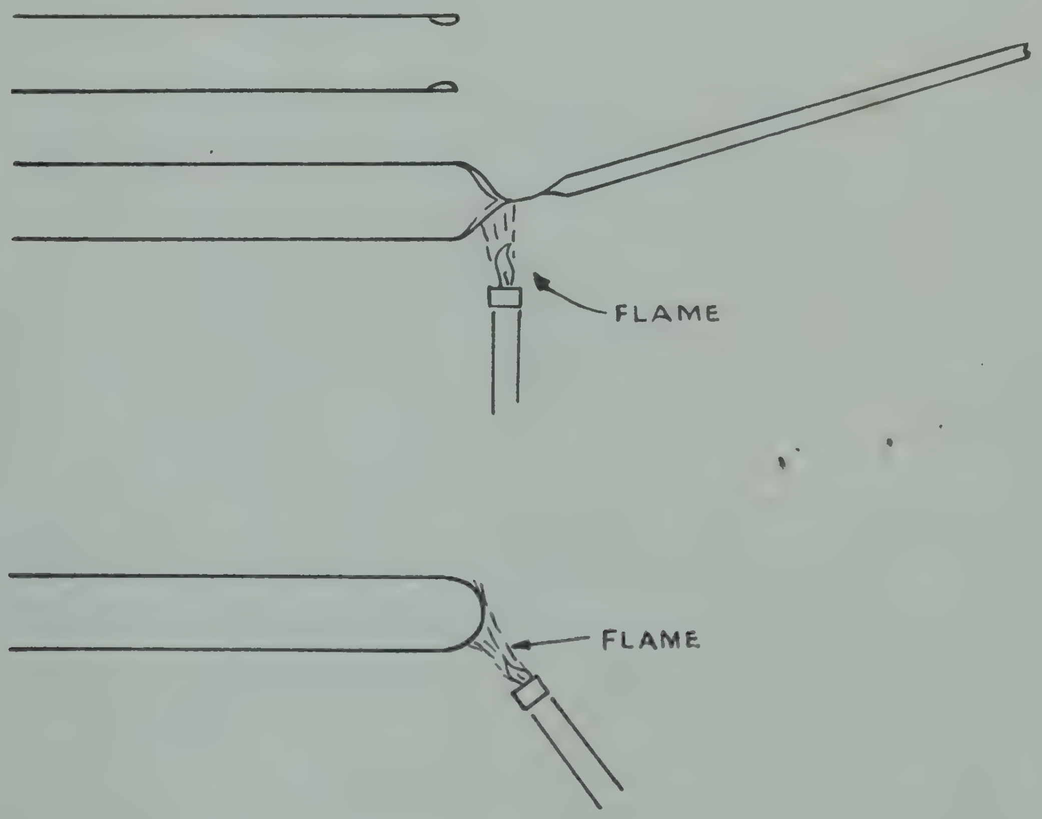

Figure 16. Round-bottomed tubes.

Flat-Bottomed Tubes

Flat-bottomed tubes are started by following the procedure for making round-bottomed tubes, with the exception that the blow tube is not used. After the round end is formed particular attention should be paid to removing the lump of thick glass at the center of the rounded section as completely as possible. When removal of this thick spot is complete the tube should be rotated with the flame played directly at the center of the tube, as shown in Figure 17B. This will cause the end of the tube to flatten out, and by alternate raising and lowering of the flat end of the tube as shown in Figure 17C, the distribution of the glass in the flat section will become uniform.

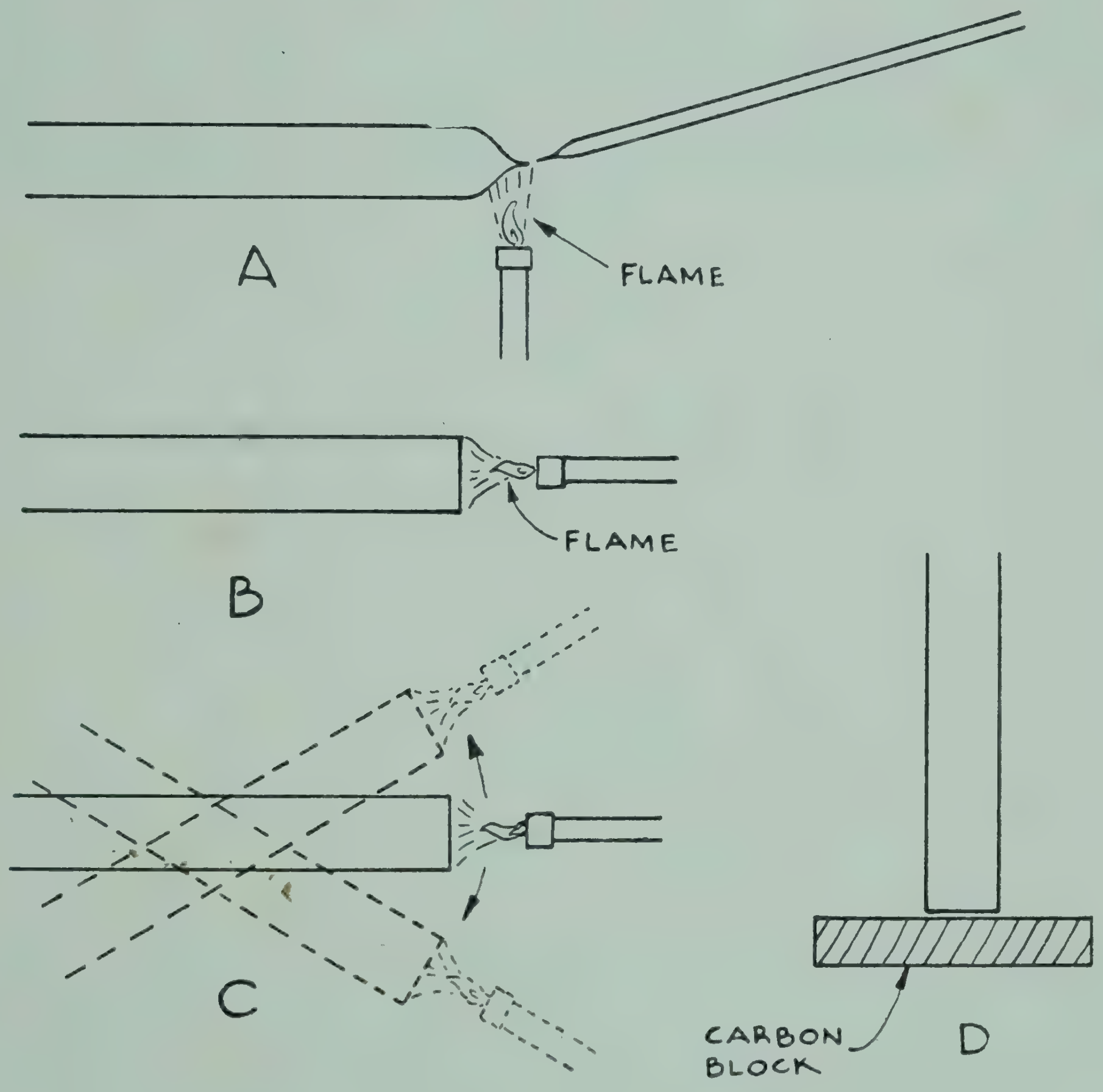

Figure 17. Flat-bottomed tubes.

Care should be exercised to see that the flame is not too large, since too large a flame will splash over the edges of the tubing, thus softening too much glass and resulting in uneven edges and a flat surface not at right angles to the axis of the tubing. Special care should be directed towards selecting a piece of tubing with as even a wall thickness as possible for making flat-bottomed tubes. The time spent in going over and checking the glass tubing to be used will be repaid by smoother, better looking work.

A flat-bottomed tube can be made by resting the heated end of a closed tube on a carbon block as shown in Figure 17D; however, the flattened section made this way will not have the optical clearness or smoothness obtained by the first described method.

Making Constrictions

Three types of constrictions can be made, as shown in Figure 18A, B and C.

In the first the outside diameter of the tube remains the same but the inside bore is constricted. This type of constriction, shown in Figure 18A, is widely used in such apparatus as manometers to slow the flow of mercury. It is stronger mechanically because of the heavy walls; however, because of these heavy walls, it should be well annealed as soon as it is finished. To make this type of constriction, a glass tube is rotated while being heated in a gas-oxygen flame. During this heating a slight compressing pressure is maintained between both hands, thus building up the wall thickness. No blowing is necessary if the heating is done carefully, but it is recommended that a blow tube be attached for use in case the heating is done too rapidly. Rapid heating will cause the glass to shrink too fast and result in the inside bore's being smaller than desired, or with extreme heating the tubing will close up solid, resulting in a section of rod. Should blowing become necessary in making a constriction, the actual blowing

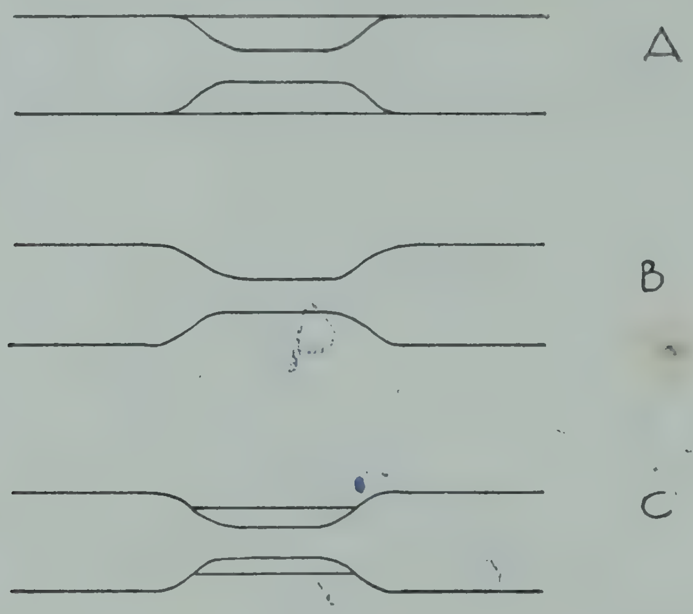

Figure 18. Constrictions.

should be delayed, after removing the glass from the flames, until the thin shoulders of the glass have time to harden. If the blowing is done too soon, these shoulders will blow out even thinner and become unsuitable for use, from the standpoint of both mechanical strength and appearance. Thin glass at the shoulders may be rethickened by carefully heating and building up the wall thickness by the same compressing pressure as was used during the first blowing of the constriction.

The second type of constriction (Figure 18B) is made by maintaining the wall thickness of the constriction the same as that of the original tube. This is done by rotating the glass tubing in a flame until it becomes soft; since a sharp oxygen-gas flame tends to thicken glass, a softer or gassy flame is used and the glass heated only to the softening point. The constriction is drawn while the glass is rotated in the flame, this drawing being continued until the constriction is nearly of the desired inside bore and length. The final drawing is done after the glass has been removed from the flame, and is just sufficient to bring the constriction to the desired dimensions and realign it with the original tubing. The use of a blow tube is also recommended in making this type of constriction, but instead of a pressure blown into the glass a slight reduction of pressure is maintained by sucking lightly into the blow tube to increase the speed with which the constriction may be drawn.

The third constriction, shown in Figure 18C, is the type commonly used for seal-off tubes of vacuum equipment. The wall thickness of these constrictions is usually two to three times that of the original tube. With the exception of a hot oxygen-gas flame used to induce the glass to thicken, the technique of making these constrictions follows that used in making constrictions shown in Figure 18B.

Healing Cracks in Glass

Healing cracks in glass tubing follows no set rule, since the success or failure of such an operation depends on what happens to the crack during the initial heating. When a completed and intricate apparatus has developed a crack, it is good procedure to test the section of glass around the crack with a polariscope to see if any serious strains remain. If this test shows that there are strains, the piece should be run through the annealing oven before attempting to make the repairs.

With smaller pieces of apparatus and those not yet finished, preliminary running through the annealing oven is not necessary. With these and with a crack running lengthwise down a tubing healing may often be accomplished by heating the tubing below the end of the crack very carefully. As the temperature of the glass approaches the temperature of the flame, the burner is slowly moved, bringing the heat closer to the crack. A very gassy flame should be used for this initial heating with very little if any air turned on until the glass has been heated sufficiently for the flame to be played directly on the crack. In some cases the gas-air flame alone creates enough heat to close the crack, although usually it is safer to heat and rework the glass where the crack was with a gas-oxygen flame to assure complete fusion of the glass. If at all possible, it is desirable to blow into the apparatus during the heating to further ensure complete healing of the crack. When blowing is not possible, in some cases the glass can be sealed and reshaped with a carbon rod or paddle.

Some cracks have a tendency to run and open when a gas-oxygen flame is directed on them. To alleviate this tendency it is advisable to apply a slight sucking pressure through the blow tube when heating the glass, since this tends to pull the glass in, and thus together. When the crack does widen in spite of all precautions, a tungsten needle may be used to push the edges toward each other and so fill up the crack, or glass cane may be used and the crack actually filled with fresh glass. When the latter method is used, it is important that the glass cane be clean and free from dust or foreign material which might be worked into the glass.

Peeling or Wiping

The method by which a jagged or uneven edge of a glass tube is made square by heating it in a sharp gas-oxygen flame and pulling excess glass off with a glass rod is called peeling or wiping.

To do this a short segment of the rough edge is heated until soft and the softened glass pulled away by means of a glass rod or a pair of tweezers. It is better to heat and pull a small amount of glass at a time than to try to remove too large a section at one time. The usual difficulty experienced by most beginners is caused by heating too much glass or heating the edge of the tube to too great a depth. This will cause too large an area of glass to become soft and pull or thin out, still leaving an uneven and unsightly end. The best results will probably be achieved if the glass is heated to a depth of not more than $^1/_{16}$ in.

Use of Glass Cane or Patch Rod

One of the advantages of glass as a material with which to work is the ease with which all traces of mistakes and accidents may be removed. It is, of course, true that a careful worker who accurately prepares all parts to be assembled together will end up with the most professional appearing work. But it is equally true that with the judicial use of patch rod (cane), apparatus which would otherwise be discarded can be made as good as new.

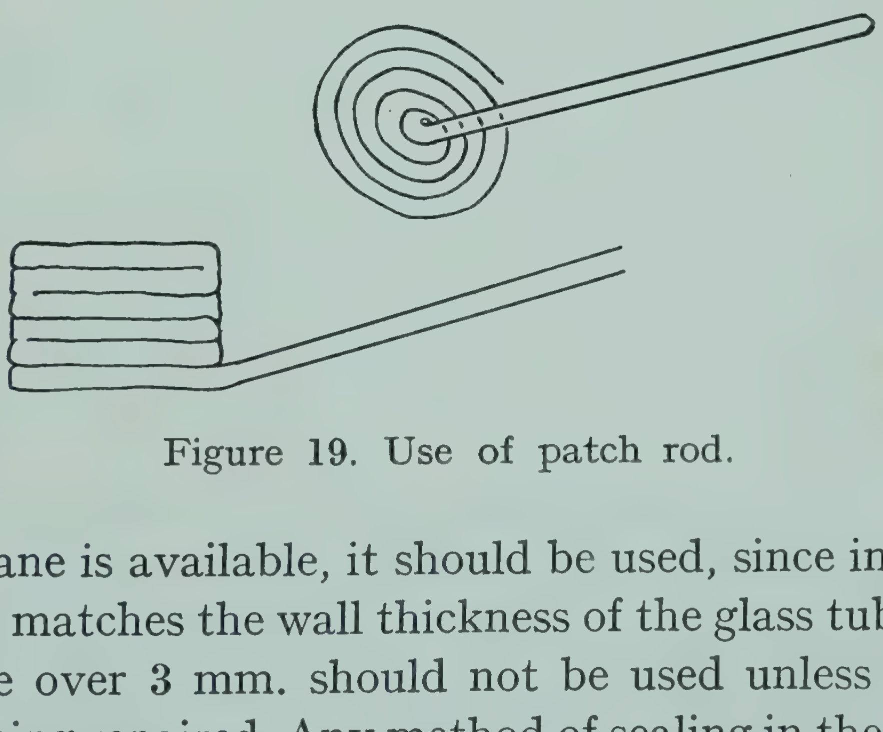

Figure 19. Use of patch rod.

If 2-mm. cane is available, it should be used, since in most cases this closely matches the wall thickness of the glass tubing. In any event, cane over 3 mm. should not be used unless heavy wall tubing is being repaired. Any method of sealing in the rod may be used, but it is important to keep the flame small so that only the immediate section of glass and cane to be fused is heated. Figure 19 shows two designs for fusing glass cane in a hole, but any pattern or combination of patterns which allows the cane to be filled in without overlapping is acceptable.

V

FUNDAMENTAL GLASSBLOWING SEALS

INTRODUCTION

Since glassblowing consists largely in sealing glass tubes together, the straight or butt seal should be thoroughly mastered by the beginner before any additional work is considered. From three to four practice sessions of 2 hours each should be sufficient for this.

BASIC OPERATIONS

Straight Seals

In making a straight seal, the tubing is held in both the right and left hands (as described in Chapter IV under "Rotation of Glass"). The glass in the left hand is rotated, with the tip of the tubing held in the flame. At the same time, the tubing in the

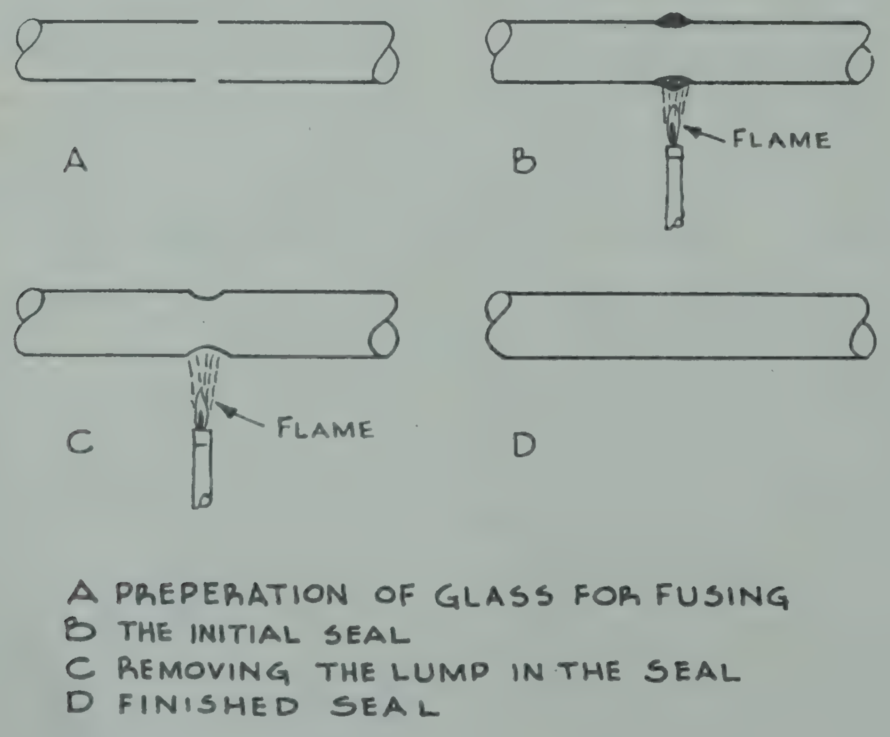
A
B
FLAME
C
FLAME
D
A PREPERATION OF GLASS FOR FUSING
B THE INITIAL SEAL
C REMOVING THE LUMP IN THE SEAL
D FINISHED SEAL

Figure 1. Straight seals.

right hand is rotated backward and forward in a 60° arc with the tip of the glass in the flame. When both pieces of glass are hot and before they shrink out of shape, they are removed from the flame and pressed together. After it is certain that they have been sealed completely around the circumference, the seal should be pulled slightly and a puff of air directed into it. The last two operations of pulling and blowing should be done without hesitation, since Pyrex solidifies quickly after being removed from the flame. If the seal appears quite thick, the wall thickness may be brought more in line with that of the original tubing by pulling, as shown in Figure 1C. When the drawn section has hardened, the glass at the seal is reheated by rotating the glass in the flame until it is pliable, after which it is removed from the flame by lowering the left hand and raising the right hand, with the true axis of the tube maintained in the process. The rotation is continued in this position and the seal pulled slightly, while at the same time occasional puffs of air are directed in the tube until the seal is of the same diameter as the original tube.

Points To Remember in Making Glass Seals

(1). Any smoothing out or dispersing of lumps in the glass must be done while the seal is being heated in the flame. (2). No seal should be worked too long. Overworking a seal tends to emphasize the irregularities in the original seal. This is especially true for beginners. More actual skill is attained by making many seals quickly than by spending a long time on a few seals.

Seals between Tubing of Different Diameters

These can be made two different ways. In both methods, the large piece is prepared as in making a round-bottomed tube. The draw-off tip is not fused smoothly into the rounded end but is reheated and blown out as shown in Figure 2A. This should be blown out to form tubing of the same diameter as the piece to be sealed on. This may be done by setting the flame as shown in Figure 2A. The seal may then be made as with two pieces of tubing the same diameter; the finished seal is shown in Figure 2B. Care should be taken to see that the flame does not play upon the round section of the large tube and distort it. The second method differs only in the last step and in the setting of the flame. Both pieces of glass tubing are prepared as before, however. In this case the flame is directed on both the large and small tubing, as shown in Figure 2C. Both pieces are rotated while being heated in the flame and a light pressure inside the glass is maintained through the blow tube. The small tubing in the right hand is pushed gently towards the left hand; this will cause the small tube to expand, working the actual seal into the rounded portion of the large tube as shown in Figure 2D. The first method will probably be easier for the beginner; however, the second gives a more professional appearance and the glassworker should practice both methods until satisfactory results can be achieved with either one.

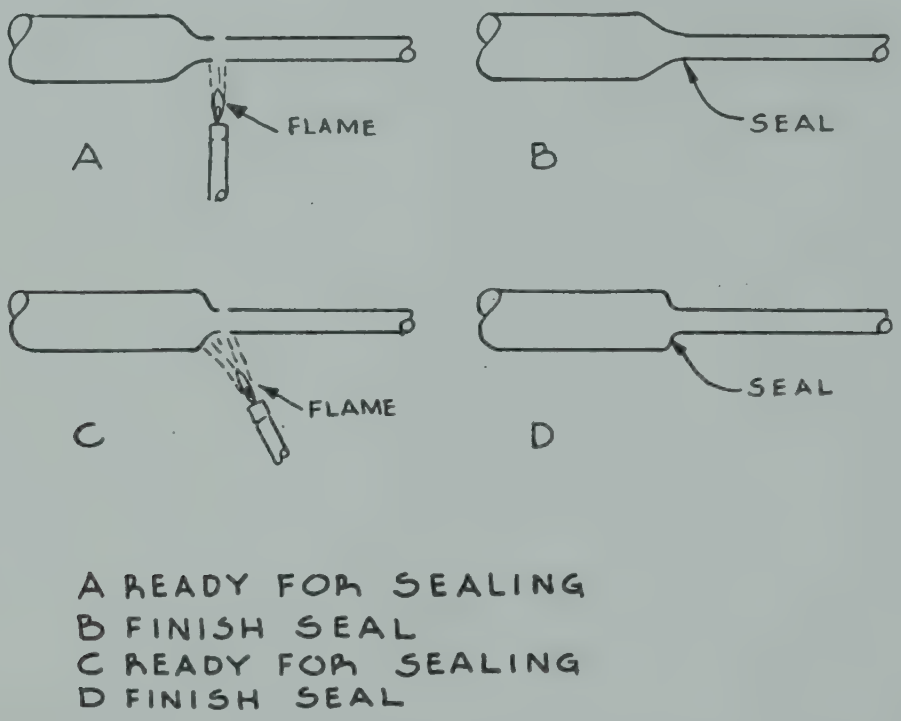

Figure 2. Seals between tubes of two different diameters.

T-Seals with Small Tubing

A T-seal is essentially a butt seal that is not easily rotated unless a holder is available. To make a T-seal, one end of the tube is closed with a cork and a blow tube attached to the open end. A hand torch is used to produce a sharp flame, and a spot is heated on one side at the middle of the tube. When the glass is heated sufficiently, a bulge should be blown as shown in Figure 3A. For tubing up to 16 mm. O.D. this bulge should be of approximately the same size as the tube to be sealed on. This bulge is then reheated with a sharp hot flame and a thin-walled bulb blown (Figure 3B). After this bulb is broken and the broken edges smoothed with a file, the open hole should be about the size of the tube to be sealed on and preferably should be slightly smaller. The size of the hole is controlled largely by the size of the area heated before blowing. If the hole is too small, the glass should be reheated and the hole reamed out with a carbon rod. However, it is desirable to get the hole as near to the right size as possible by blowing. In making the seal, the edge of the opening and the end of the tube are heated uniformly until they are soft. They are then removed from the flame and brought together quickly, and pulled slightly as soon as contact has been made while at the same time light blowing is used to expand the glass and help remove any irregularities (Figures 3D). If the glass was sufficiently soft when it was joined, this procedure will produce a good seal. If the joint does not appear uniform, it should be spot heated with in a sharp pointed flame and the glass melted to work out irregularites and bring the seal into proper shape. The seal should be annealed with a gas-air flame when it is finished and all the angles between the tubes brought into adjustment.

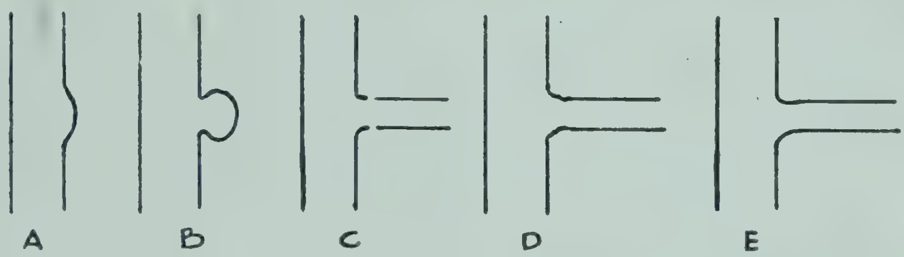

Figure 3. T-seals in small tubing.

T-Seals by Rotation

A 6- to 8-in. piece of Pyrex rod may be fused to a glass tube about 5 in. long, as shown in Figure 4A. The tube is heated exactly opposite the attached rod and a tip is drawn at this point by means of a second glass rod held in the right hand, as shown in Figure 4B. This tip is reheated and blown out, as shown in Figure 4C. The seal may now be completed as essentially a straight seal (Figure 4D and E).

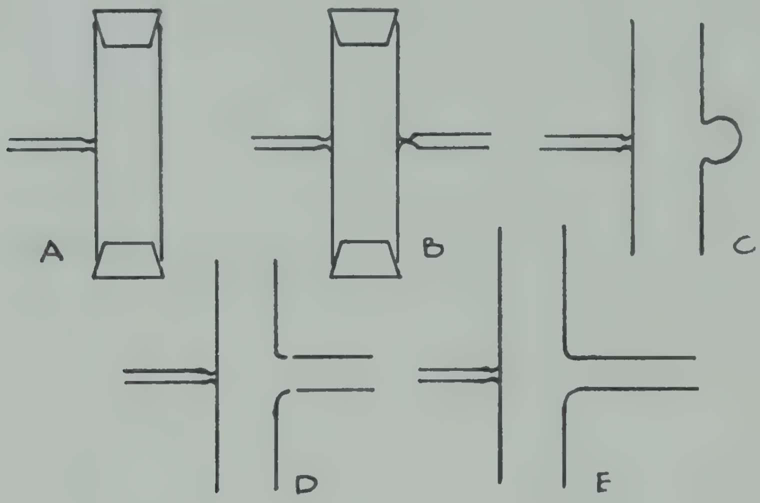

Figure 4. T-seals by rotation.

T-Seals with Large Tubing

In making a large T, the side arm should be prepared first by shrinking one end down to about three-fourths of its original size, as shown in Figure 5A. In preparing the second piece, it is advisable to preheat it before attempting to blow the hole for the side arm. This hole is prepared by following the same procedure as outlined on page 79. A bulge is blown in the side of the tube and the top of this bulge reheated and blown out. The finished hole

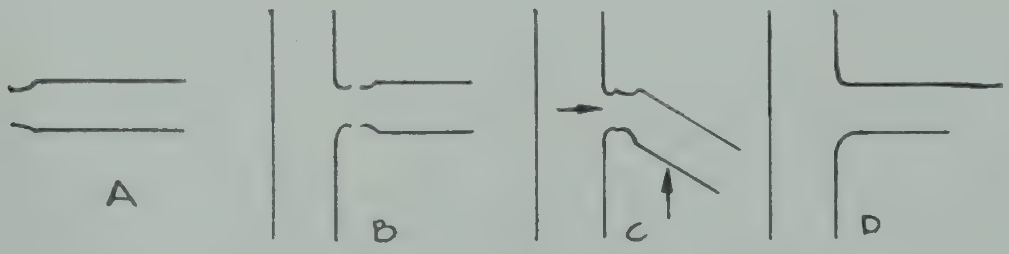

Figure 5. T-seals in large tubing.

should be slightly less than three-fourths the size of the side arm, since this side arm will shrink further on being heated. If the blown hole is too small, it is possible to enlarge it by flaring with a carbon rod; however, too much flaring is not desirable since it presents a curved surface in place of the flat face necessary for satisfactory sealing. After the necessary heating, contact is made between the end of the tube and the face of the hole with the rocking motion shown by the arrows in Figure 5C. When complete contact is assured, the seal is finished by spot heating with a sharp flame as for seals with small tubing.

Suck Seal

When it is not possible to blow into a piece of apparatus, a T-seal may be made by the suck-seal method. In this method the side arm is flared slightly and a cotton plug inserted at the opposite end. A pinpoint oxygen-gas flame is played on the second piece of glass at the spot where the seal is to be made (Figure 6A), while at the same time the side arm is warmed in the splash from the same flame. When the spot on the main tube appears to be sufficiently soft, the side arm is placed in position, pushed down to assure even contact all around, and, before the glass has time to harden, sucked on so strongly that the hot glass expands into the side arm and bursts, leaving a smooth seal. The cotton in the side arm keeps small pieces of glass from the bursting bubble from being drawn up into the mouth. If the bubble does not break but forms a very thin globe, this may be broken by removing the cotton and puncturing the thin globe or bubble with a wire. If the spot was not heated enough, or if the suck was too weak to break the bubble, it will be necessary to heat the seal and remove the side arm before proceeding with a new seal, this time using the standard procedure for side arms if possible. If the seal was properly made, the suck seal requires only annealing. This should be done immediately following completion of the seal

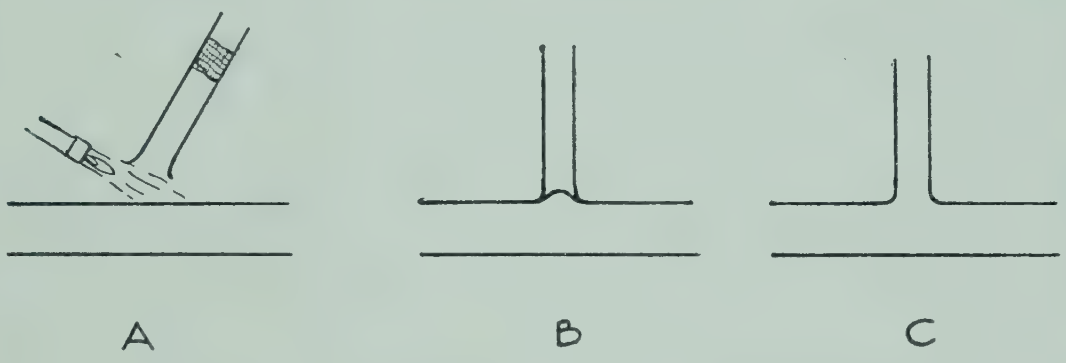

Figure 6. T-seals by the suck-seal method.

since, because of the method of construction, excessive stresses are set up in the glass during the glassblowing operation.

Ring Seals

Ring seals or supported inner seals are made as shown in Figure 7. The inner tube is first flared and then wrapped with a supporting roll of asbestos tape. This support should be of the proper diameter to form a smooth fit within the outer tube. When it is left too loose, the inner tube will move during the sealing operation, whereas too tight a fit makes it difficult to control the blowing during sealing. It is also very hard to remove the asbestos when the seal is finished. When the proper fit is obtained, either air or water or a combination of both can be used to remove the asbestos support.

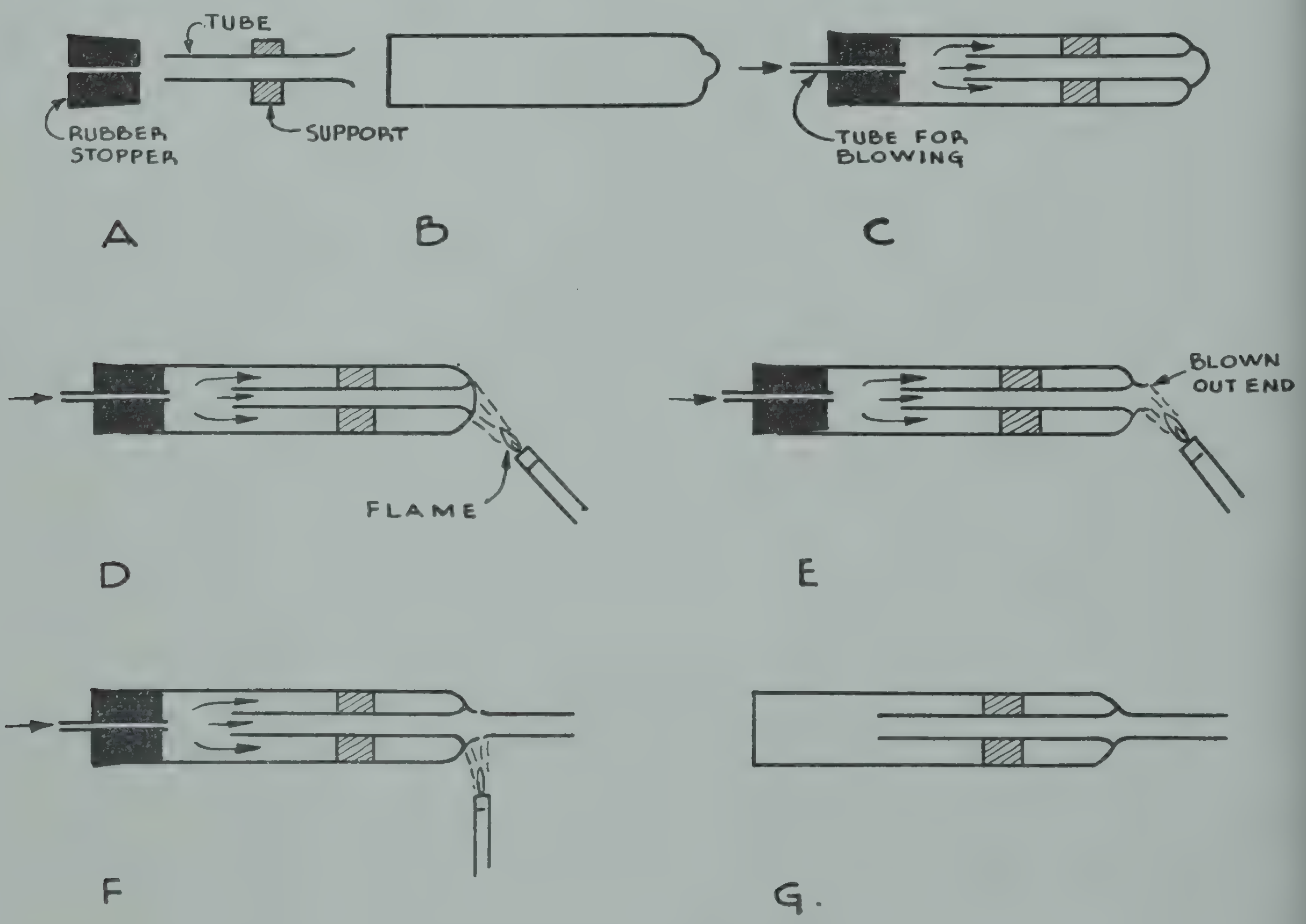

Figure 7. Ring seals.

To prepare the outer tube, it should be sealed off at one end and a small bulb blown, as shown in Figure 7B. This enlargement or bulb should be just large enough for the inner tube to fit into and center itself. In glass tubes of large diameter and for long inner tubes, two asbestos supports may be used to ensure centering the inner tube in the closed end.

When both pieces are ready, they should be assembled as shown in Figure 7C, and the flame adjusted to play over the end of the large tube (Figure 7D). The glass is worked by alternately blowing slightly and allowing it to shrink back until a smooth ring seal is clearly visible. When this ring can be seen, the flame is reduced and only the center of the small tube is heated. When the glass is sufficiently soft at that point, a few gentle puffs of air will blow out a small bulb. This bulb is reheated and the gentle puffs of air should be continued until the bulb is of the same diameter or slightly larger than the tube to be sealed on. When the proper size bulb has been reached, the glass is allowed to cool and then the bulb is reheated and blown out (Figure 7E). The seal may then be finished as in making a straight seal. At times it is more convenient not to remove the interior support until the apparatus is completely finished. When this is to be done, copper screen (page 38) should be used in place of asbestos tape.

Insertion Seals

Insertion or unsupported ring seals are made as shown in Figure 8. The inner tube is prepared first by heating a small section in a sharp flame, and then blowing and pushing slightly to form a bulge, often referred to as a "Maria" (shown in Figure 8A). The diameter of the bulb or Maria should be from 2 to 4 mm. larger than the tube itself.

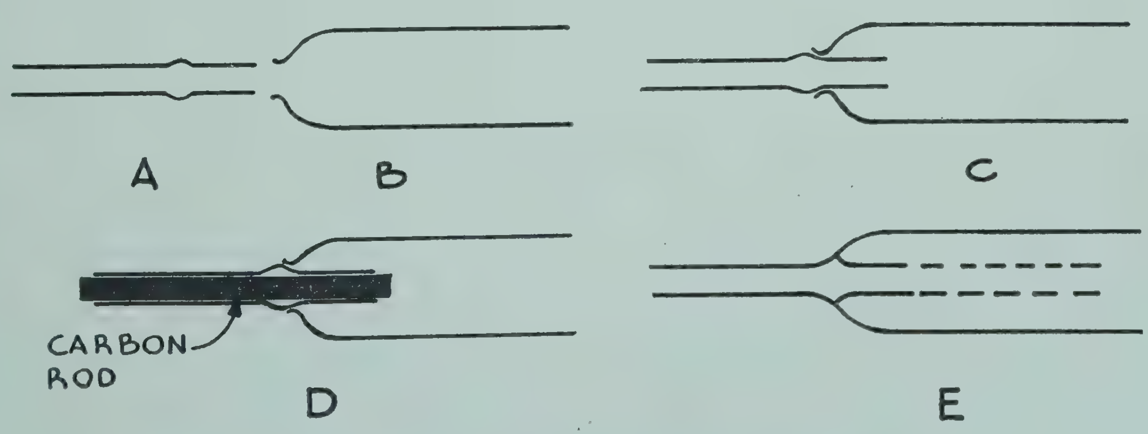

Figure 8. Insertion seals.

The large tube may be prepared by making a test tube end and blowing a small hole out of it as shown in Figure 8B. This hole should be of the proper diameter to enclose the bulb on the small tube (Figure 8C). If the inner tube is short, no support will be needed, since even rotation will hold it centered. Longer inner tubes may be supported with asbestos (see page 82, "Ring Seals") or with a carbon rod which is inserted through the small tube (Figure 8D). During the operation of making the seal, a flame is directed onto the small tube and the shoulder of the large tube as in making a seal between tubing of different diameters (page 77). If the inner tube is out of line, it may be centered if the seal is reheated and the glass rotated, bringing the inner extension in line. When the inner section is correctly lined, the outer section may be straightened by reheating and moving it in line; care should be taken to avoid heating the shoulder and moving the inner tube out of alignment again. The completed seal is shown in Figure 8E.

Dewar Seals

These seals consist of two glass tubes, one inside of the other, but sealed together at one end without an extension coming through as with a ring seal. The seal may be made several different ways.

Method I. The inner tube is flared at one end and made into a round-bottomed test tube, the proper length required for the Dewar. The outer tube is closed at one end as in making a ring seal. Supporting asbestos tape is wrapped around the inner tube and the two assembled as in Figure 9A. Continuing as in making a ring seal, the end is heated until the two tubes are sealed together. This leaves the inner tube without an outlet and the expanding hot air inside causes the heated glass to expand into a bubble (Figure 9B). Continued heating and removal of this expanding glass with a rod will create a bubble so thin that it will finally break. When this happens the excess glass can be flared until it is of the same diameter as the inner tube and then removed by peeling or wiping (page 74). The remaining glass may then be worked into a smooth seal by continued heating, rotating, and paddling with a carbon rod. During this heating the burner should be set at right angles to the glass and the flame pointed directly at the seal. A carbon flaring tool is used to prevent the inner tube from becoming smaller, and an occasional puff of air will prevent the outer tube from shrinking out of shape. The occasional puffs of air will gradually cause the inner tube to creep out until the actual point of seal is in the position shown in Figure 9C. The seal is improved in appearance by being reheated again and by having the inner tube pushed back, with the aid of a long carbon rod, until the point of seal is located as shown in Figure 9D. As a final step, the Dewar is reheated and the carbon flaring tool is used to bring the inner tube to the correct diameter and the end is paddled with a carbon plate to assure a flat face; care should be taken in the meantime to avoid blowing the glass enough to bring the seal back out over the lip.)

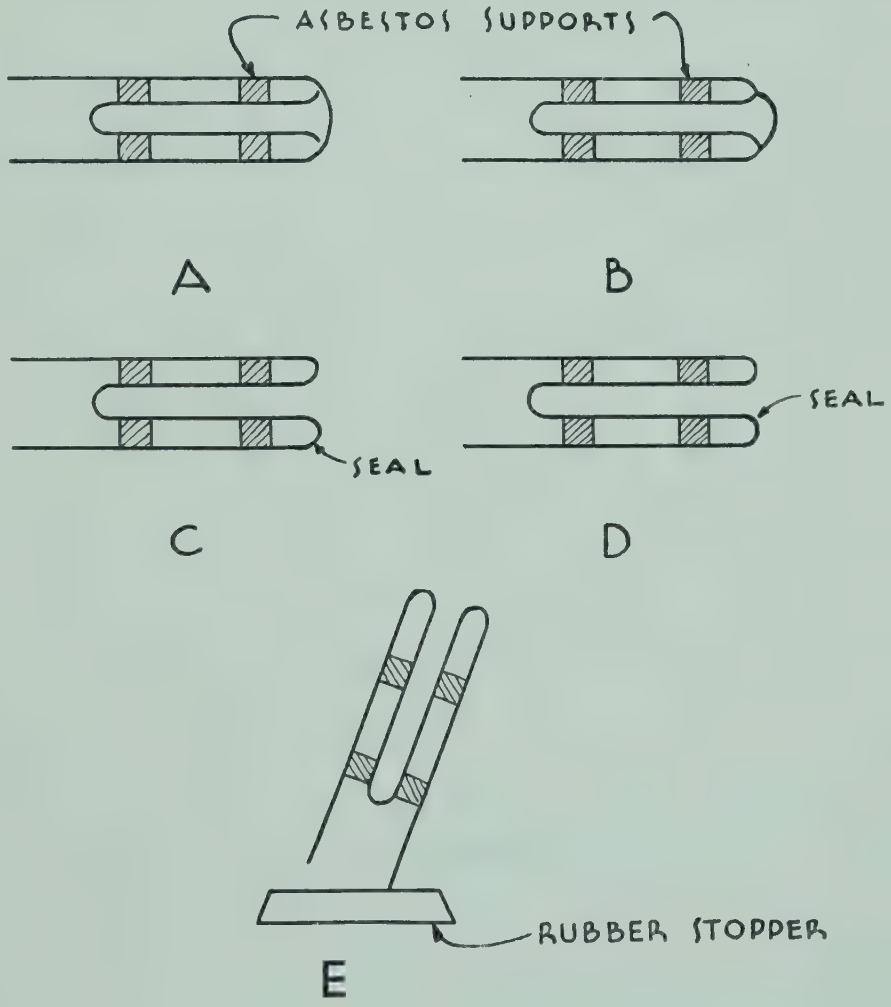

Figure 9. Dewar seals, method 1.

If a side arm is to be added near the Dewar seal, it should be sealed in position and the finished end thoroughly annealed. In the case of Dewars with a side arm, the asbestos supports may be removed by means of water and compressed air. Dewars that are to have no side arm should have the asbestos supports wrapped loosely enough that they will slide out when dampened with water. Tapping the Dewar on a rubber stopper (Figure 9E) will help remove supports which are wrapped correctly. If the asbestos has been wrapped too tightly, a $^1/_{16}$ in. brass rod bent to form a hook may be used to pierce the wetted asbestos and remove sections at a time.

Copper screening may also be used as the means of supporting the inner tube, and may be removed with nitric acid later.

Method 2. It is sometimes possible, especially in large Dewars, to leave a small hole in the bottom of the inner tube for blowing through (Figure 10A). When such a hole is used, the two tubes are assembled and the seal started as in making a ring seal. After the end has been blown out, a cork or asbestos plug is inserted in the blow hole and the Dewar completed as in Method 1. When the seal is finished, the blow hole is closed by filling it with Pyrex rod and heating the whole end of the inner tube with the flame from a hand torch until the glass is fused (Figure 10B).

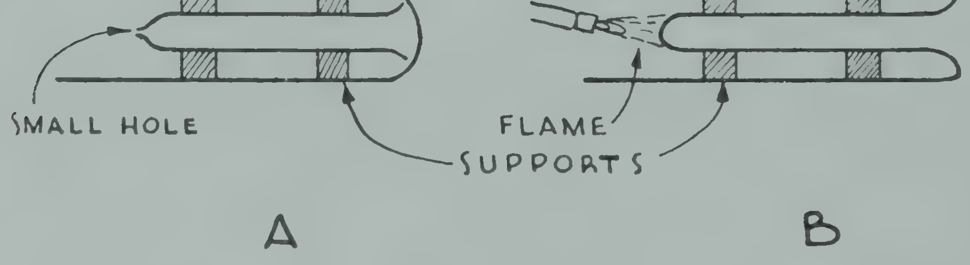

Figure 10. Dewar seals, method 2.

Method 3. The inner tube is prepared by flaring it at right angles until the diameter of the flare is the same as the diameter of the outer tube. After the end opposite the flare is given a test tube bottom, the two pieces are assembled, as shown in Figure 11A, with the flame striking the outer tube edge. By rotating the tubes with a flame focused at the points of contact, a carbon rod or carbon plate can be used to bring the two edges together to form a seal. If the edges are uneven, it may be necessary to use a little glass rod to close small holes during the initial joining. A combination of flaring the inner tubing and shrinking the outer tube (Figure 11B) may also be used, and the seal made as just described.

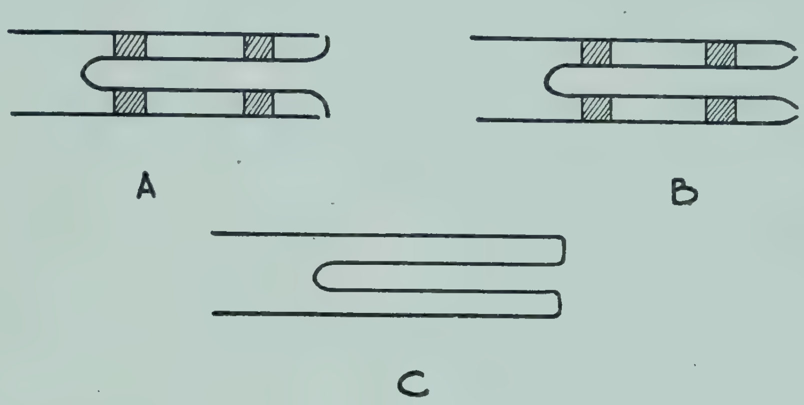

Figure 11. Dewar seals, method 3.

Bulbs at the End of Glass Tubing

When special apparatus is built, standard commercial flasks can usually be used for the various sizes of bulbs needed. In many cases, however, and especially when small bulbs are needed, it is advantageous for the glassworker to make the bulb himself.

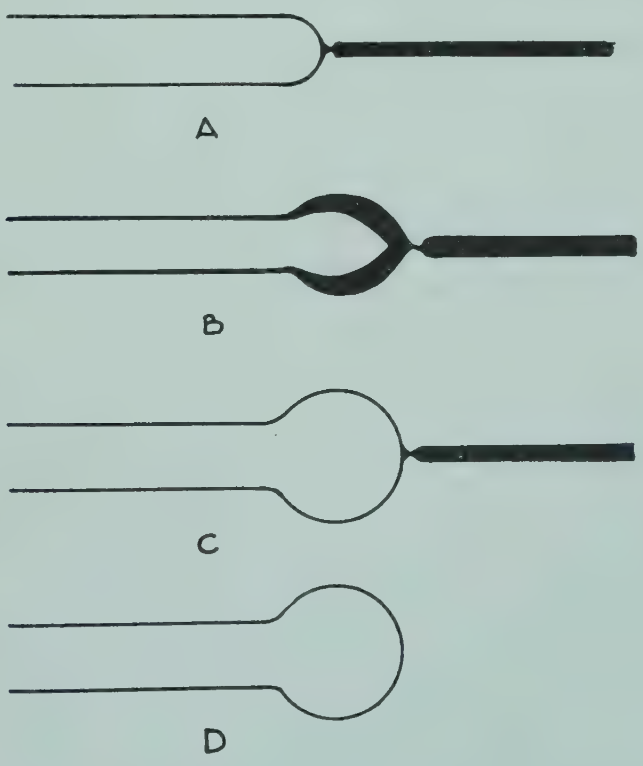

Figure 12. Bulbs at end of tubing.

Small bulbs less than 50 mm. in diameter can be made from tubing by the following procedure. The end of a piece of tubing approximately half the diameter of the required bulb is closed off and a rod handle attached as in Figure 12A; when this is ready, the glass may be heated preparatory to blowing the bulb. The length of tubing heated depends upon the size of the bulb and the wall thickness required. During heating, a section of glass is shrunk to about half its original length while the full diameter is maintained with occasional puffs of air through the blow tube (Figure 12B). When the thickened tube is quite soft, it may be removed from the flame and blown to the required diameter using the rod in the right hand as a guide for keeping the bulb in line with the axis of the tube (Figure 12C). Uniform rotation is imperative in order to obtain uniform heating and cooling. Without it, the glass will flow unevenly because of gravity. The bottom of the bulb is finished by removing the rod used for the handle and treating the bulb in the same way as for making a round-bottom tube (Figure 12D). In blowing very small bulbs, a handle is not necessary, since even thickness of the wall can be obtained by uniform rotation and heating.

Bulbs in the Center of a Section of Tubing

Considerable skill and dexterity are required in rotating the glass tubing while the necessary glass for the bulb is being accumulated in the middle of the tube. A blow tube is connected to the special stopper which is held in the left hand, and a glass rod handle is sealed to the other end. The amount of glass required for the bulb will depend on the diameter and wall thickness desired. Gathering the glass for the bulb may be done several ways.

Method 1. A section of glass tubing is rotated in a flame until it begins to shrink. A light pressure is applied to the inside of the glass through the blow tube, to hold the diameter constant while the glass is thickened by a light compressive pressure maintained between both hands. When enough glass has been thickened, it should be removed from the flame and blown into slightly, as shown in Figure 13A. This operation is repeated adjacent to the blown bulb, and for larger bulbs this operation may be repeated a third time (Figure 13B). The thickened sections of glass are then heated in a bushy flame until all sections of the glass have an uniform wall thickness (Figure 13C). The glass is then heated again and blown out as shown in Figure 13D.

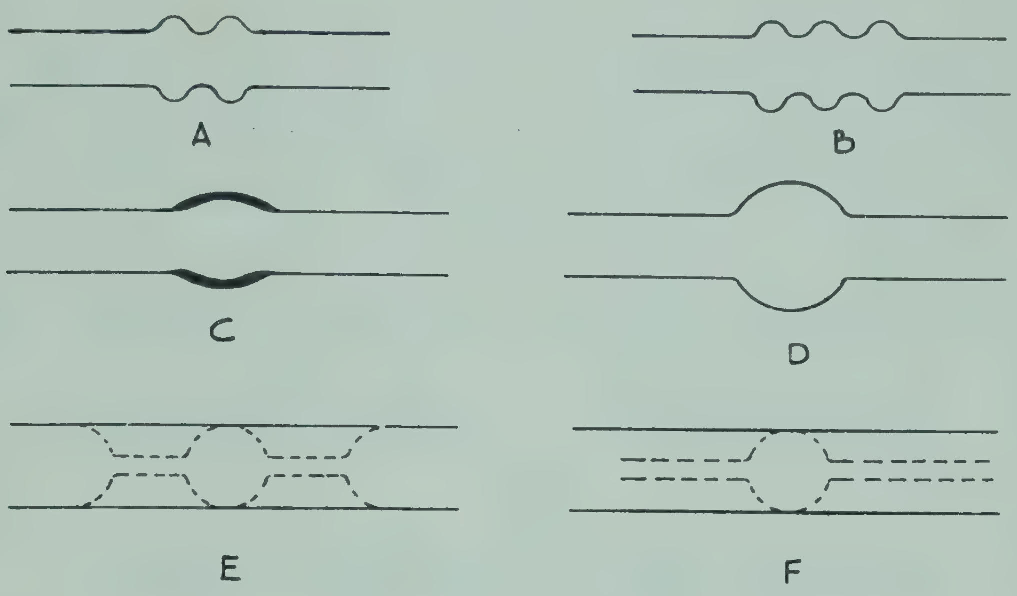

Figure 13. Bulbs in center of tubing.

Method 2. For many bulbs the first step of the above operation can be continued until enough glass has been collected in the original gather to make the bulb, thus eliminating the second and third steps. When this method is used, the bulb is gradually enlarged while in the flame, until it is about three-fourths of the desired diameter. When this approximate diameter is reached, the completed bulb is heated evenly just before being removed from the flame and being blown out to the desired size. The appearance of bulbs made with one gather will be freer from the lines which are often visible in bulbs made with several separate gathers.

Method 3. Bulbs may be made by using glass tubing of the same diameter as the finished bulb and shrinking sections of the glass at intervals to form the connecting tubing between bulbs (Figure 13E), or, alternatively bulbs may be made by making a closed round bottom on a piece of glass tubing the required size, after which a side tube or neck of the desired diameter is sealed in place and this operation repeated on the opposite side of the bulb (Figure 13F).

The skill required in handling large sections of heated glass places a definite limitation on the size of the bulb blown. Blowing uniform bulbs is one of the more difficult operations of freehand glassblowing, but proficiency itself, when attained, is a satisfactory reward for the time and effort spent in practicing.

Sealing Capillary Tubing to Standard-Wall Tubing

Capillary tubing which is to be sealed to standard-wall tubing is prepared by heating the end, pulling off a section of glass by means of a glass rod, and blowing the closed end into the shape shown in Figure 14A. This end is then reheated and blown into a bubble having the same O.D. as the capillary tubing and the approximate I.D. of the standard-wall tubing (Figure 14B). The top of this bubble should then be heated and blown out as shown in Figure 14C. The excess glass is removed by breaking the thin bubble with a file. The end is reheated again and shaped with a carbon rod, care being taken not to distort the capillary tube itself (Figure 14D). The seal with the standard-wall tubing may now be completed as in a straight butt seal; again care must be exercised to keep the flame away from the heavy capillary walls. If necessary, the burner should be turned so the flame is played in the direction of the standard-wall tubing (Figure 14E).

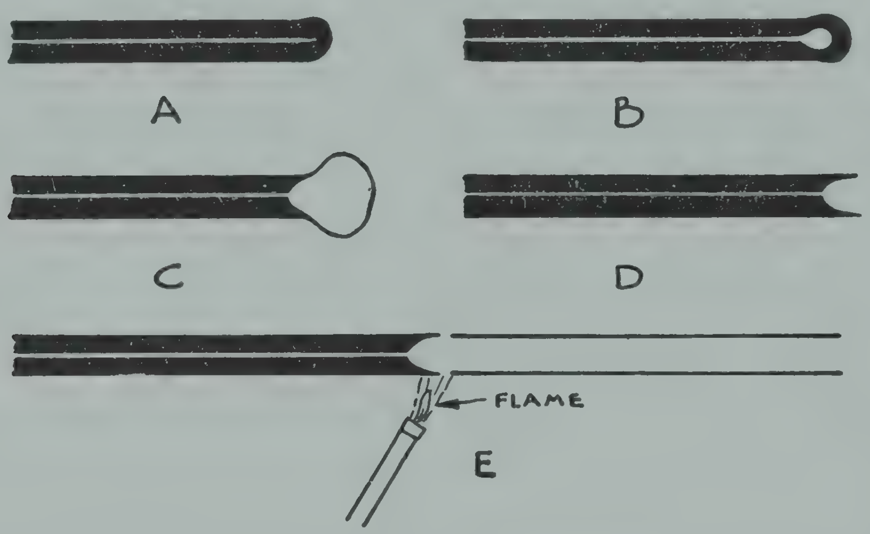

Figure 14. Capillary seals to standard-wall tubing.

Capillary T or Side Seals

The capillary T or side seal varies from the standard T mainly in the care that must be taken not to shrink the inner bore of the tubing. The side arm is prepared in the same manner as when making butt seals to standard-wall tubing. The other section of the T is prepared by preheating it in a gas-air flame.

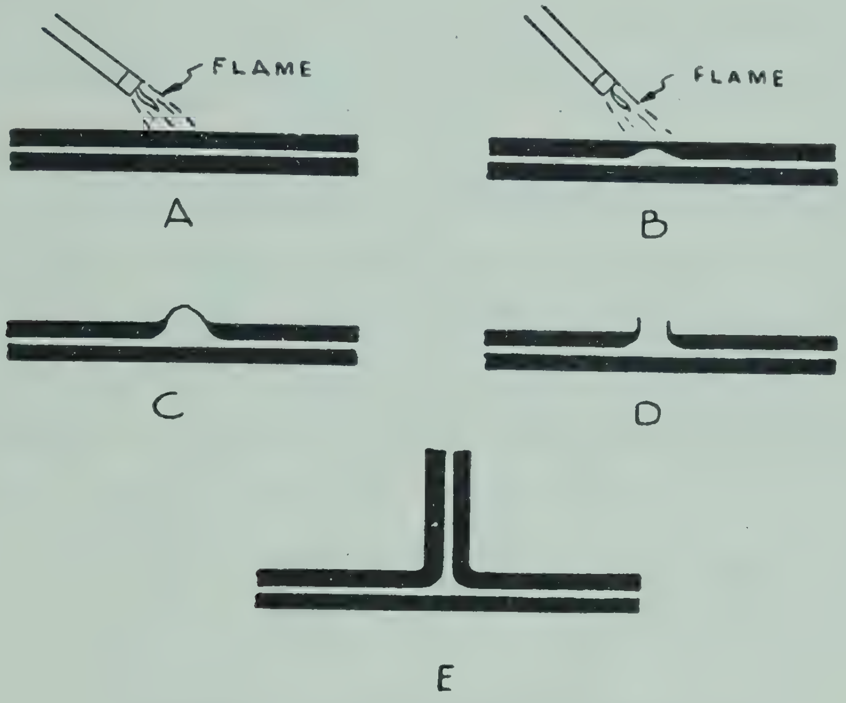

Figure 15. Capillary T-seals.

When the glass has been heated through, a sharp, hot oxygen flame is directed on one side at the point where the seal is to be made.

An occasional puff of air through a blow tube will indicate when the glass is sufficiently soft by the formation of a small bubble. When the bubble begins to show in the capillary wall, a strong *steady* pressure should be maintained inside the tube until the bubble assumes the appearance shown in Figure 15B. This spot is reheated again and blown until the bubble (Figure 15C) is formed. The top of the bubble should be reheated and blown out and the thin excess glass removed with a file. The edges may then be thickened as shown in Figure 15D. The actual seal is made by heating the glass with a small pointed flame, eliminating flame splash and the danger of deforming the internal diameter of the capillary (Figure 15E).

Bubbles may be blown in capillary tubing in one operation. Better results will usually be obtained, however, with the controlled step method just described.

If the glassworker has difficulty keeping the different arms in alignment, he may find it easier to lay the T on a piece of clean Transite and to use the spot method, heating first on the top and two sides, then turning the T over and repeating the operation on the other side. It is important that the glass be kept hot during the sealing operation to prevent cracking, and a capillary T should be annealed immediately upon completion of the seal.

Sealing Two Pieces of Capillary Tubing

Making a butt seal between two pieces of capillary tubing is similar to sealing two pieces of standard-wall tubing except that greater care must be taken in aligning the inner bores when they are first placed together. A small, hot flame is used to reduce flame splash with the resultant heating of too large an area. Heating of too large an area deforms the inside diameter of the capillary and results in a seal of poor appearance.

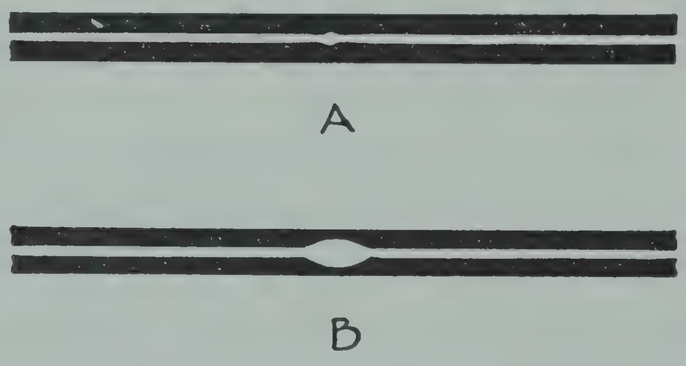

Figure 16.
Straight seals between two pieces of capillary tubing.

Figure 16B illustrates a seal made by flaring both pieces of capillary tubing before sealing them together.

Bends

The diameter of the tubing to be bent, and the distance desired between the tubes after the bend, will determine the length of tubing which must be heated. The end of the tube to be held in the right hand, is closed with a cork and the blow tube is attached to the other end (left hand). The tube is rotated in the flame, heating a sufficient length necessary for the bend. Two procedures are used:

Method 1. The tubing is rotated, while it is held in the hand, the same as for making straight seals.

Method 2. In the second method of heating, the tube is not actually rotated. Instead it is heated between cross fires, turned 90°, and heated again. By alternately heating, turning forward 90°, heating, and turning backward 90°, all four sides may be heated evenly and a satisfactory bend can be made. When the tube has become pliable, it is removed from the flame and quickly bent upward, allowing the heated section to drop downward. As soon as the bend is completed and the tubes are parallel, air should be blown into the bend with sufficient pressure to remove the irregularities in the bend and also to expand it to the original diameter of the tube. (See Figure 17.) *Notes:* 1. Blowing into the tube before the bend is completed will cause it to bulge.

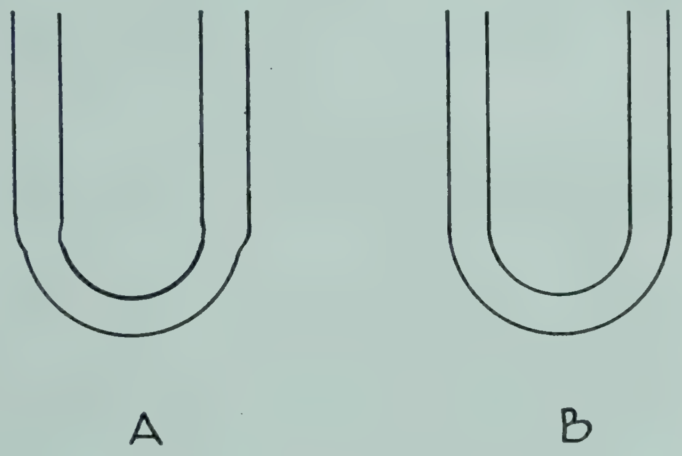

Figure 17. Bending glass tubing.

2. Pushing the tube together or stretching it during heating will make it more difficult to obtain a smooth even bend. With larger sizes of tubing (25 mm. and up), it is desirable to shrink the tubing to as much as three-fourths of the original diameter before making the bend and then to blow it back to its original size.

Right-angle (L) bends are made by following the same procedures. Bends are not usually made in a horizontal plane unless a template is being used to form a definite arc.

Coils from Tubing

Coils may be made freehand but only an expert can make presentable coils in this manner. Satisfactory coils are made quickly and easily on carbon mandrels which can be purchased in many different diameters from the National Carbon Company. A hole is drilled at one end of the carbon rod (see Figure 18) for holding the glass tubing. The equipment necessary to make coils consists of one roller support stand, one ribbon burner, and a carbon mandrel of the desired diameter. In adjusting the burner flame for winding coils, it is well to remember that sufficient heat to soften the glass is all that is required, and this amount of heat may be obtained from a flame that is 80 % to 85 % gas, with just enough oxygen to produce a small blue oxygen cone at the base of the flame. (See Figure 19.) With this relatively cooler flame, softening of the glass will be sufficiently slow that the glassworker has time for turning the mandrel, blowing, and spacing the coils evenly. With a properly adjusted flame the worker will find himself able to wind coils rhythmically. Rotate the mandrel enough to add ½ to ¾ in. of glass tubing to the coil, puff of air through blow tube, rest, rotate mandrel, puff of air,

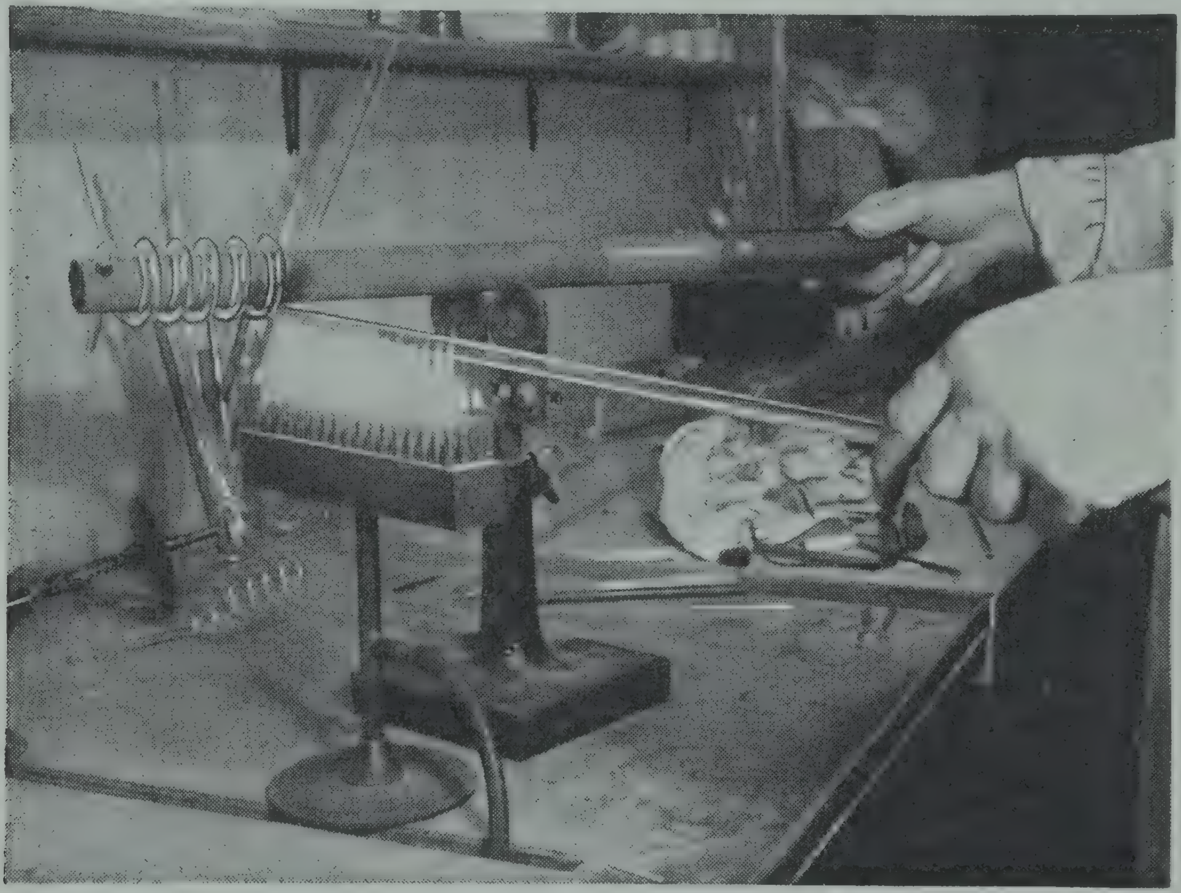

Figure 18. Winding glass coils.

rest, etc. After each turn of the mandrel, a sharp puff of air will return the glass tubing to its original round shape again, ready for the next turn of the mandrel. The slight pause or rest allows time for the next section of glass to soften sufficiently to be bent around the mandrel. The worker should experiment with different settings of the burner flame, until a flame is found that will allow him to perform the three steps in the rhythm of a slow 1—2—3 count. By varying the burner flame this same rhythm may be used for either large or small tubing regardless of the diameter of the mandrel.

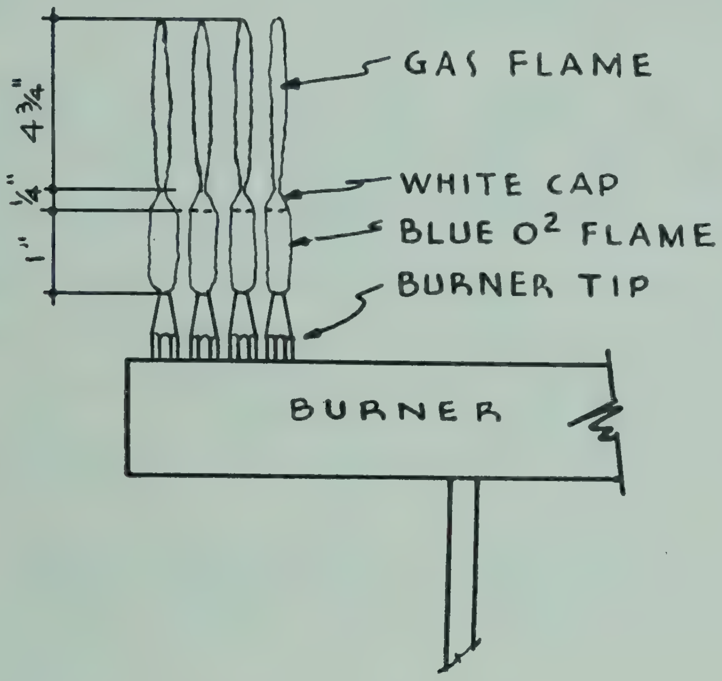

Figure 19. Adjusting burner for winding coils.

During the operation of winding coils the importance of the left hand being held steady cannot be overemphasized. The left hand controls the speed of feed of the tubing onto the mandrel. When the left hand pushes the glass tubing onto the mandrel faster that it is being wound, unevenly spaced coils will result, while if the left hand is slow and pulls or stretches the tubing, the finished coils will have turns of uneven diameter tubing and will be unsightly. The even spacing between the coils is controlled by the angle at which the mandrel is held in relation to the position of the fires. Once set, this is maintained evenly by the steady winding of the tubing on the mandrel.

Metal Mandrels. When carbon mandrels are not available, metal ones may be used; however, it is advisable to make some sort of a heat-resistant handle before attempting to use a metal mandrel. Large-diameter coils can best be wound on metal mandrels and these should be warmed before winding the tubing in order to prevent chilling the glass, which might cause it to crack.

Other Mandrels. Glass tubing may also be used for a mandrel, provided it is first wrapped with wet asbestos paper, which is smoothed at the seams and allowed to dry thoroughly. A general practice with asbestos-covered mandrels is to make the mandrel one day, allow it to dry overnight, and make the coil the next day. Since the asbestos cover is usually ruined in removing the coil, a new asbestos covering must generally be made for each coil.

Automatic Coil Winder. When a large number of coils are to be made, the coil winder shown in Figure 20 is very useful. By the use of collets, carbon rods of different diameters may be used. By loosening a wing nut, the angle at which the carbon rod is held can be changed, thus changing the pitch of the coil. A ribbon burner may be held in the left hand and played on the tubing while the right hand slowly turns the mandrel. For tubing up to

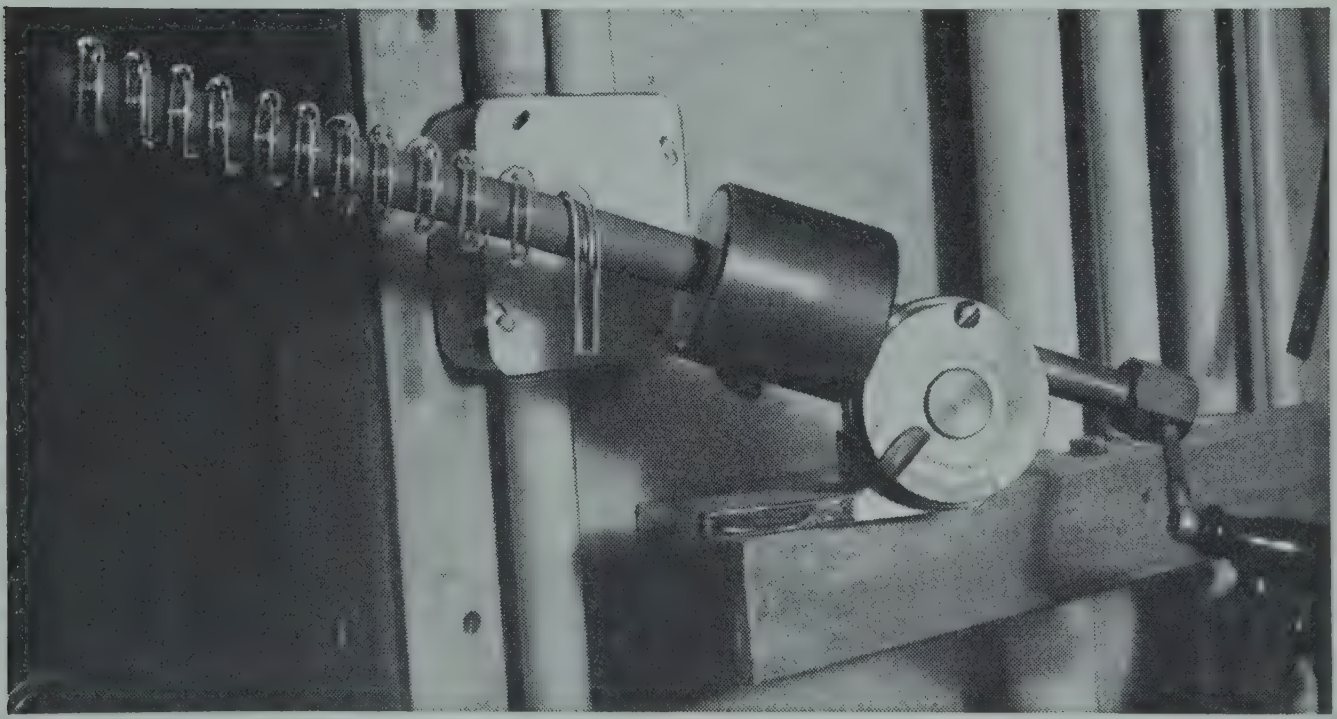

Figure 20. Automatic coil winder.

8 to 9 mm. blowing will not be necessary to produce an even coil. Tubing above 8 mm. can be wound by attaching a blow tube to the end hanging down and blowing and turning with the same rhythm employed in making coils, as described in the preceding paragraph.

Side Ring Seals

A frequently used seal is one in which a small tube is brought out through the side wall of a larger tubing (Figure 21). The inner tube is prepared first by making a right-angle bend and cutting it to the proper length to fit against the outside tube when supported in position (Figure 21A). The assembly should be such that the glassworker can blow either into both the large and small at one time, or into only the small tube by itself. In making the seal, the area of the outside tube is heated at the point where it makes contact with the inner tube until the glass is fused sufficiently to form a seal. During this heating, the blow tube is attached so that pressure is applied simultaneously in both tubes. After a good seal is obtained at the juncture of the inner and outer tubes, the blow tube is attached to the inner tube only, the seal reheated and blown out to a small bulb which, in turn, is reheated and blown out to a thin-walled bulb. After the excess glass is scraped away and the opening fire-polished, the side arm is added as in making a T seal. The large tube should be heated in a Bunsen flame periodically to prevent its cracking during construction

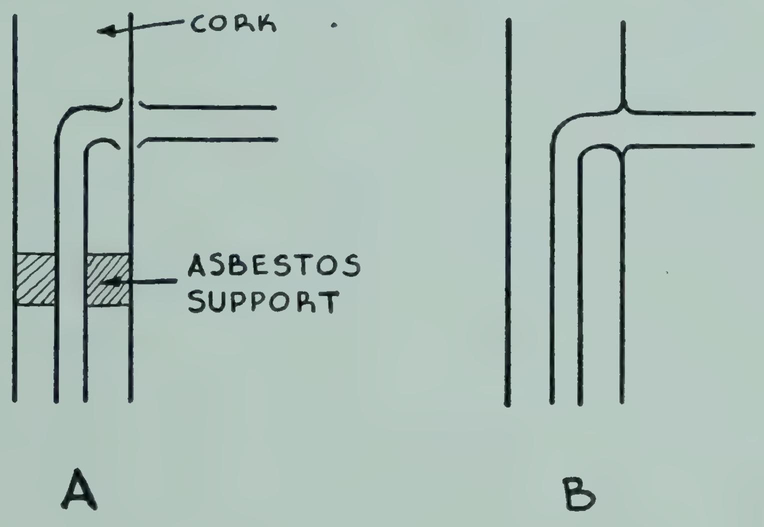

Figure 21. Side ring seals.

and should be thoroughly annealed immediately on completion of the seal. The asbestos support can be removed by wetting it and tapping the glass gently but firmly on a piece of cloth on the glassblowing bench. If the finished apparatus has a side arm, removal of the asbestos is simplified, since air or water can be used to remove it.

Closed Circuits of Tubing

In many cases it is necessary to seal both ends of one tube to another tube. This can be done in several ways, two of which will be discussed here.

Method 1. The tube to be sealed on is bent or shaped to the desired measurements. The two spots where seals are to be made on the tube are carefully marked and a bulb is blown at each of these two points (Figure 22A). One bulb is reheated and blown out, and the two tubes are sealed as shown in Figure 22B. The second bulb may then be heated and blown out, after which the first seal is reheated and the tube moved into position. This juncture is then heated and worked out smooth; any holes opening up may be closed with either a tungsten needle or patch rod. When this second seal has been finished and annealed, the first seal should first be warmed, then reheated and worked smooth before being thoroughly annealed. The first seal should be nearly at room temperature before the second is given the final annealing. If it is not sufficiently cool, stresses set up when both seals become cool may cause a crack in the closed circuit.

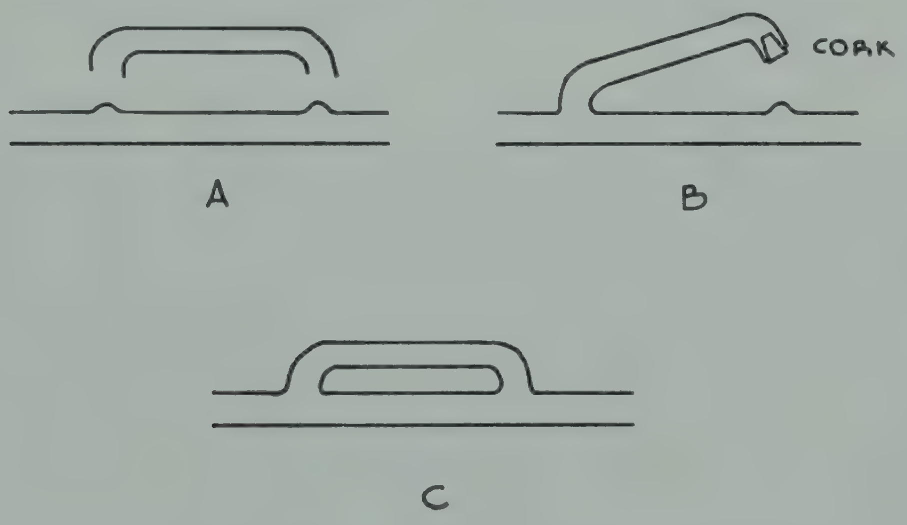

Figure 22. Closed circuit seals, method 1.

Method 2. Two tubes are first prepared as shown in Figure 23A and a seal made in the usual manner. Using a sharp flame, the tube is blown open at the point where the other end should be attached. The small tube may then be cut, allowing sufficient length for the bend (Figure 23B). The opening and the end of the tube are heated until they are quite soft; at the same time the flame is played on the tube where it is to be bent. With a pair of tweezers, the tube is bent and the heated ends forced together. A sharp, hot flame is used to fuse the glass and smooth the seal to a uniform size and shape. The glass should be carefully annealed after each seal is finished. A third version of this method of making closed circuit seals is shown in Figure 23C.

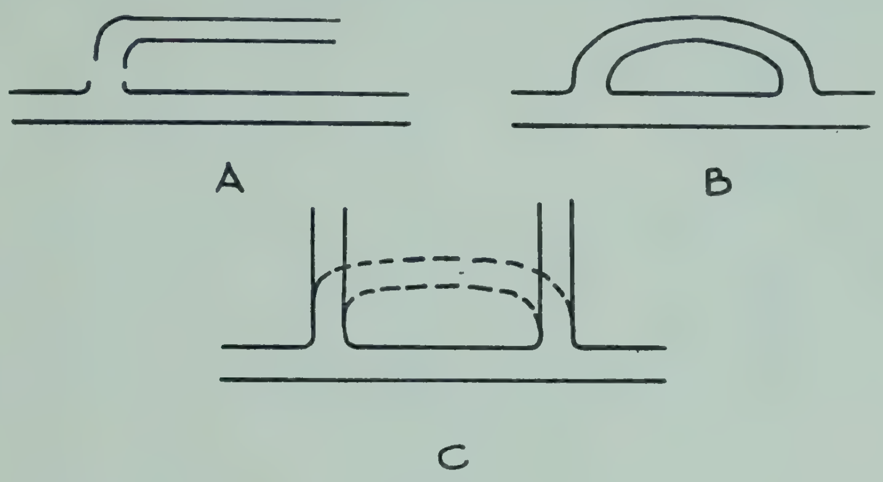

Figure 23. Closed circuit seals, method 2.

Braided Glass Coils

Double, tightly wound coils, in appearance resembling braided coils, can be made by the following procedure. A tube of the desired diameter is prepared, as shown in Figure 24A, with a ribbon burner and about the same flame adjustment used for wind-

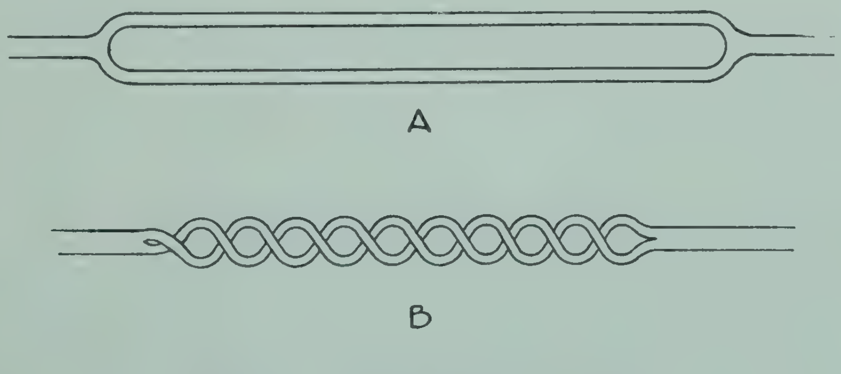

Figure 24. Braided coils.

ing standard coils. A portion of the glass is heated and twisted as it becomes pliable, with a new section gradually heated as the braid progresses. These coils should be thoroughly annealed as soon as completed to prevent possible cracking. In use, the ends of both sections are blown out and sealed to one piece of tubing as shown in Figure 24B.

Hand Torch Seals

Glass equipment which is mounted in racks, such as vacuum equipment, must of necessity be built in sections and sealed together in position, using hand torches. It is equally as important to develop the skill of making seals in which the glass is heated evenly by moving the burner as it is to make seals in a bench burner by the even rotation of the glass.

There are two different types of in-place seals to be considered, that in which both pieces of glass are clamped in place, and that in which one piece is free to move. Seals are more easily made with glass tubes in a vertical position than in a horizontal position.

Both Pieces Clamped. In this case it is of prime importance that both ends be cut off square and that if possible they be clamped with a slight pressure tending to push them together. Before starting any of these seals, the glassworker should be sure to have a supply of 2- to 3-mm. Pyrex rod on hand as well as a tungsten needle for closing any small pin-holes or gaps which may open. In either vertical or horizontal seals it is advisable to heat as small an area of glass as possible to keep the glass from flowing out of shape or thinning out because of gravity. After the seal is completed around the periphery of the tube, the danger of the glass's cracking may be greatly reduced by the use of two hand torches, one a National gas-air annealing torch, and the other the gas-oxygen burner. With the seal kept hot by the gas-air burner, a very pointed gas-oxygen flame may be safely used to spot-heat the seal which is gradually worked smooth and to the same diameter as the rest of the tubing.

One End Free. When the tubes are in a vertical position, making of these seals is simplified if the bottom tube is free. The ends of the two tubes to be sealed are heated and, when soft, the bottom tube is pressed up against the other, making the seal. The seal is thickened as shown in Figure 25A if the heating at this juncture is continued and the lower piece of glass is pushed upward. The flame should be removed from the seal and the bottom section pulled slowly downwards to even out the thick spot, followed immediately by a puff of air to bring the seal to the same diameter as the tubing. If the glass is pulled too fast or too long, the result will be similar in appearance to Figure 25B. This can be rectified if the tubing is again heated at the narrow spot and pushed upward slightly while at the same time air is blown gently into the glass until it has thickened up and regained the proper outside diameter.

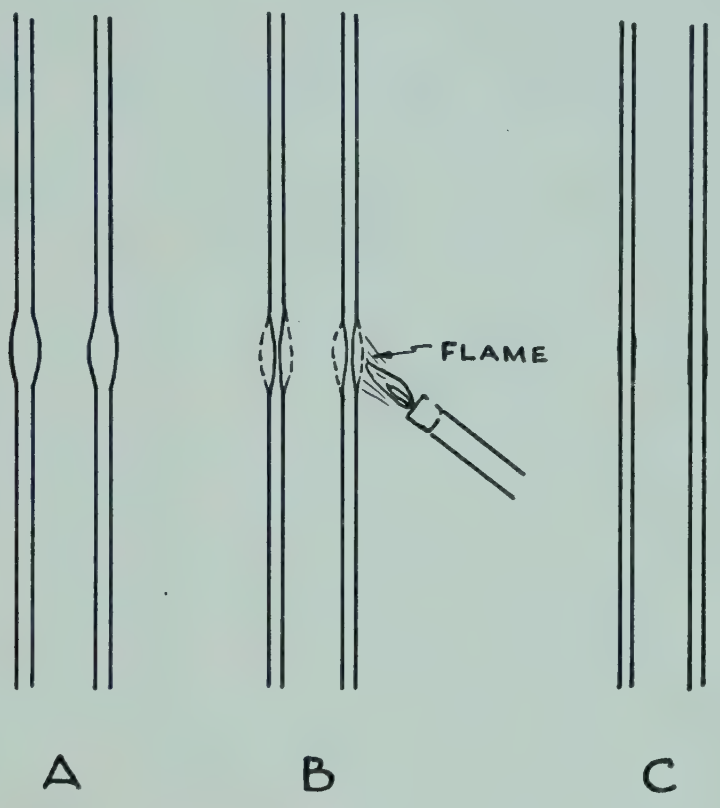

Figure 25. Making in-place seals.

Repairs to Vacuum Systems

Glassblowing around mounted vacuum systems many times calls for the replacement of a section of tubing in the system. This is made easier by the simple expedient of using cellulose tape for closing one end while performing the sealing operation on the other end. A section of tubing is cut to the proper length to fit into the system. With the tube held loosely in position, one end is heated until the glass is soft. This is then pushed against the mounted piece of vacuum system to form the first seal. Quickly, before the glass hardens, the tube is pulled until the opposite end is touching the section of the vacuum system to which it is to be sealed. A piece of cellulose tape is then wrapped around the cool ends to make an airtight joint, after which the first and partially completed seal is reheated and smoothed out. When this seal is finished, annealed, and allowed to cool, the cellulose tape is removed and the second seal is made following the same procedure described on page 100, both pieces clamped.

Sealing Glass Rods Together

When two pieces of solid rod are to be sealed together the main precaution necessary is to warm them slowly enough to prevent their cracking. Preheating with a gas-air burner is advisable. Since no blowing is possible, shaping the rod is simply done by manipulating the glass either by hand or with the aid of a carbon rod or paddle. Heated portions of glass rod must be annealed immediately after the work is finished to remove the strains and to prevent cracking.

PRACTICE PIECES TO MAKE

PLANNING THE WORK

In all but the most elementary construction jobs, it is well to form a plan of operation. This is especially true when newly designed apparatus is to be made. In addition, when intricate and complex apparatus is to be built, a planned step-by-step drawing will prove useful in preventing the operation from reaching a point at which further progress is handicapped by something left out or located in the wrong place. All parts that can be prepared in advance should be ready, as well as patch rod, tungsten needle, annealing burner, and anything that might be required in an emergency. The well-prepared glassblower seldom has the trouble that falls to the lot of the unprepared, and when trouble does come, he is able to cope with it more easily.

WORKING INSTRUCTIONS

Cold Trap

A cold trap is made following the instructions for making a ring seal (page 82) in preparing both the inner and outer tubes. When the ring seal is completed, the smaller tube is heated about 1 in. from the seal and a right-angle bend is formed. The side arm is then sealed on at an angle of 180° to the bent tube. The round-bottomed end may be made easily if the outlet or side arm is bent back to form a handle in line of axis of the cold trap. This need not be lined up perfectly with the body of the trap; however, it is good practice to bring it into as close alignment as possible. The extra step of forming the side arm into a handle can be eliminated and after some practice the end closed by rotating the trap backward and forward at a 180° angle while the excess glass is being pulled off. When this is removed, the glass is worked smooth in the flame and the end blown round with the trap held in a vertical position. (See Figure 1.)

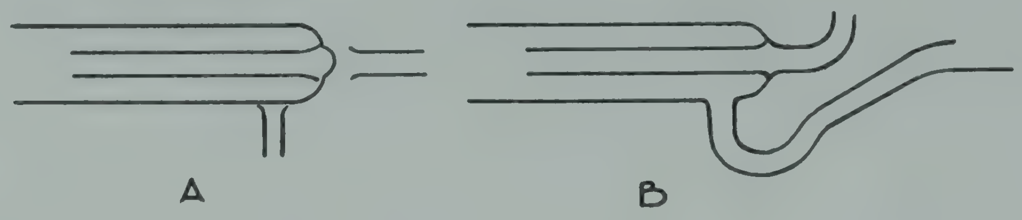

Figure 1. Making a cold trap.

Cold Trap with $ Joints

A standard cold trap for a vacuum system, and one that can be easily opened for cleaning, is shown in Figure 2B. Because the inner joint is attached to the bottom of the trap, the danger of stopcock grease working down into the trap and contaminating it is greatly lessened.

During construction, in most cases, a single roll of asbestos tape in the main body of the trap will support the inner tube while the ring seal through the outer section of the $ joint is being made. Immediately after completion of the ring seal, the smaller or through tube should be bent at right angles to the

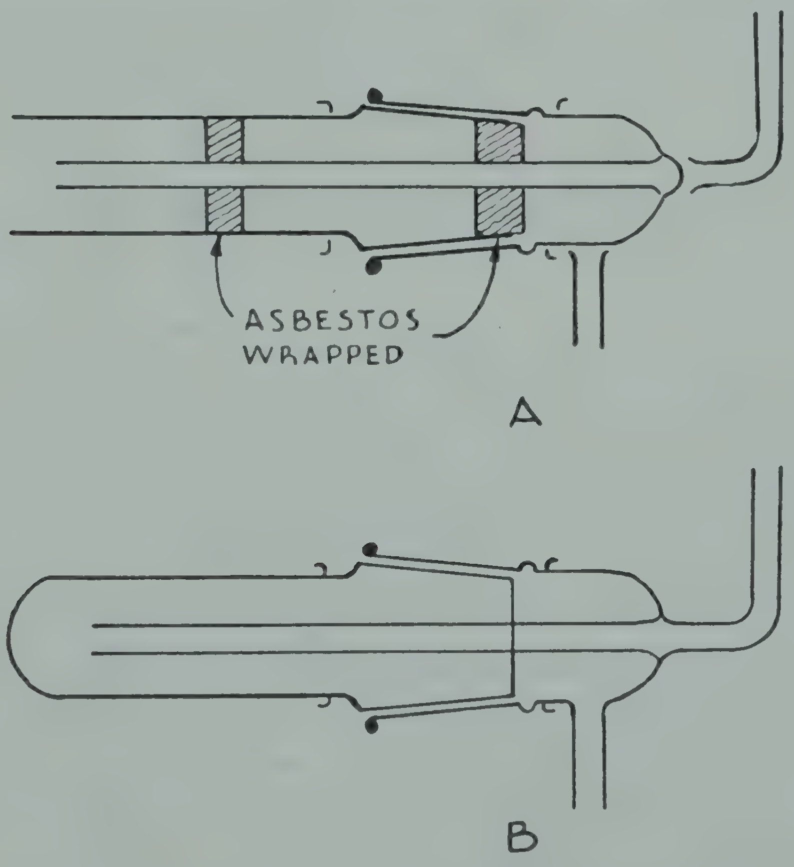

Figure 2. Cold trap made from $ joints.

main body of the trap. The inlet to the trap is then sealed on at an angle 180° to the bent tube and the completed seals annealed. Figure 2A shows the various steps in their prescribed order. When the trap has cooled, the bottom section is removed and a test tube end blown.

Standard Condenser

The basic step for making a ring seal is the starting step in making a condenser. After one end is completed and the side arm of hose connection sealed in place, this finished end is annealed and allowed to cool before the asbestos support is removed (Figure 3A). If water pressure is necessary to remove the asbestos support, the condenser should be dried before work is commenced on the other end. With the blow tube fastened to the center tube, the outer jacket is heated and pulled off to give a condenser of the required length (Figure 3B). In contrast to this method, the inner tube can be prepared beforehand by being cut to the proper length and flared, although this flaring is not absolutely necessary. When the inner tube is prepared beforehand, the outer jacket is drawn down and worked in smoothly as shown in Figure 3C.

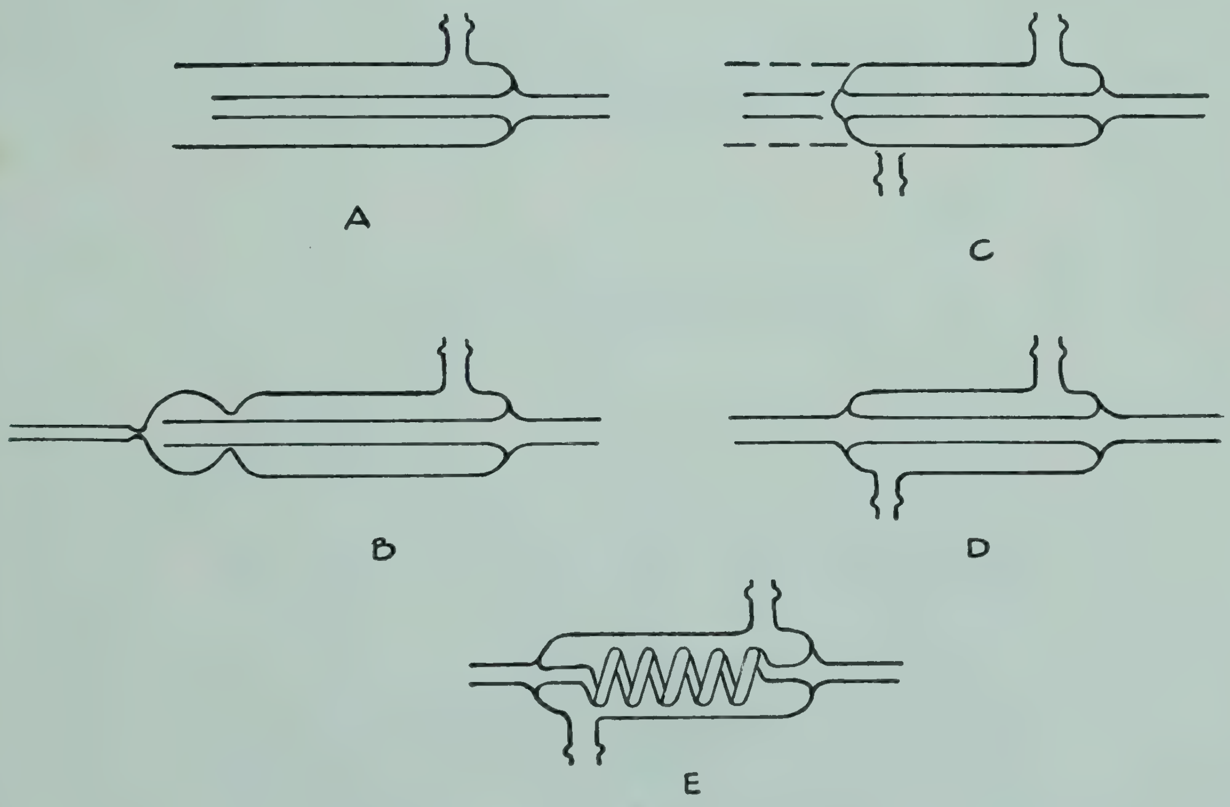

Figure 3. Standard condenser.

With the blow tube still attached to the inner tube, the end is reheated and a small bubble blown (Figure 3C). After this is blown and before the glass has time to set, a puff of air is blown into the hose nipple to round the walls at the end of the outer jacket. The seal may be completed by playing a pointed gas-oxygen flame on the bubble to blow this out to a slightly larger diameter than the tube to be sealed on. The condenser may then be finished according to the procedure for making a straight seal; care should be exercised that only the smaller tubing is heated, not the rounded walls of the jacket. Should the rounded end shrink out of shape, it may be blown back out with a quick puff of air, and the small tube may then be given the final alignment with the main body of the condenser. In Figure 3E is shown one type of standard coil condensor.

Bulb Condenser

Bulb condensers when they are not too long may be made with an asbestos support at only one end. The opposite or unsupported end is centered in an enlargement on the round bottom.

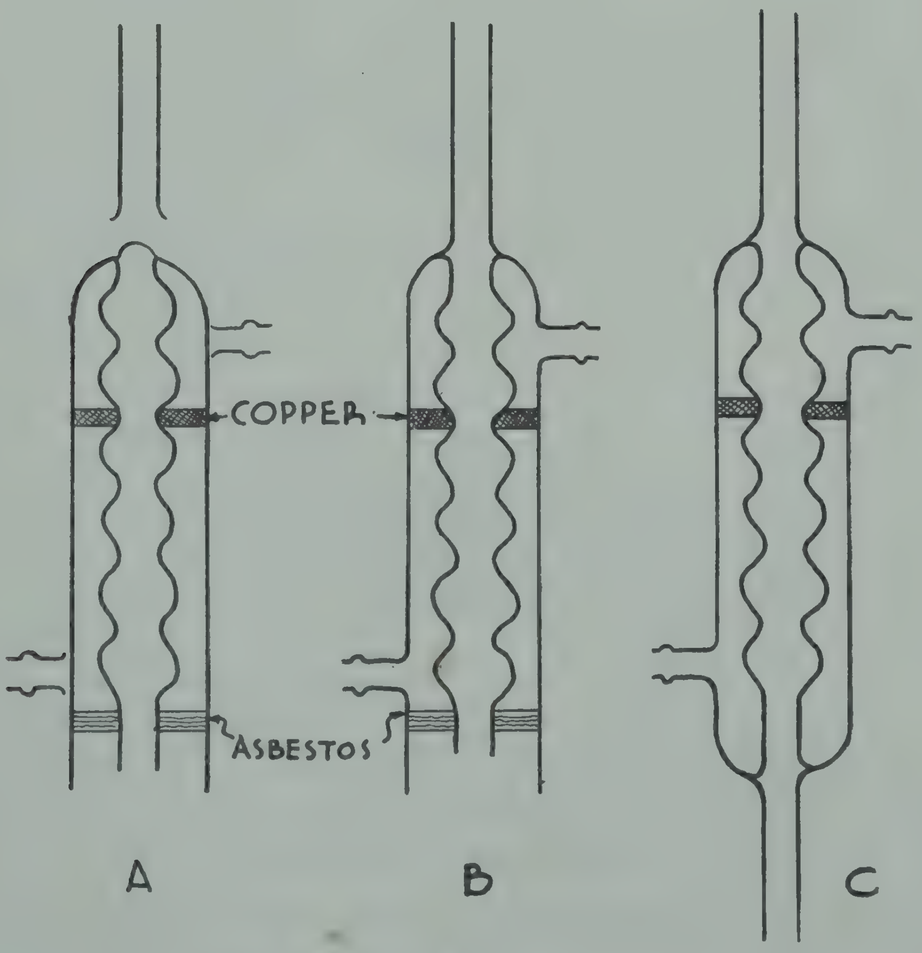

Figure 4. Bulb condenser.

By avoiding heating the round shoulders, the inner bulb section is sealed to the jacket and the smaller tube blown out as in the preceding paragraph. With some practice, even this single support may be eliminated in the smaller condensers and the inner tube lined up by rotation. Larger bulb condensers will be made more easily if two supports are used during construction. Copper screening that may be removed when the condenser is finished makes a satisfactory second support and is placed as shown in Figure 4A. The opposite end is finished as described in the preceding paragraph.

Coil Condensers

Straight coil condensers are made following the instruction for bulb condensers; however, the extremely useful condenser shown in Figure 5A requires a different technique.

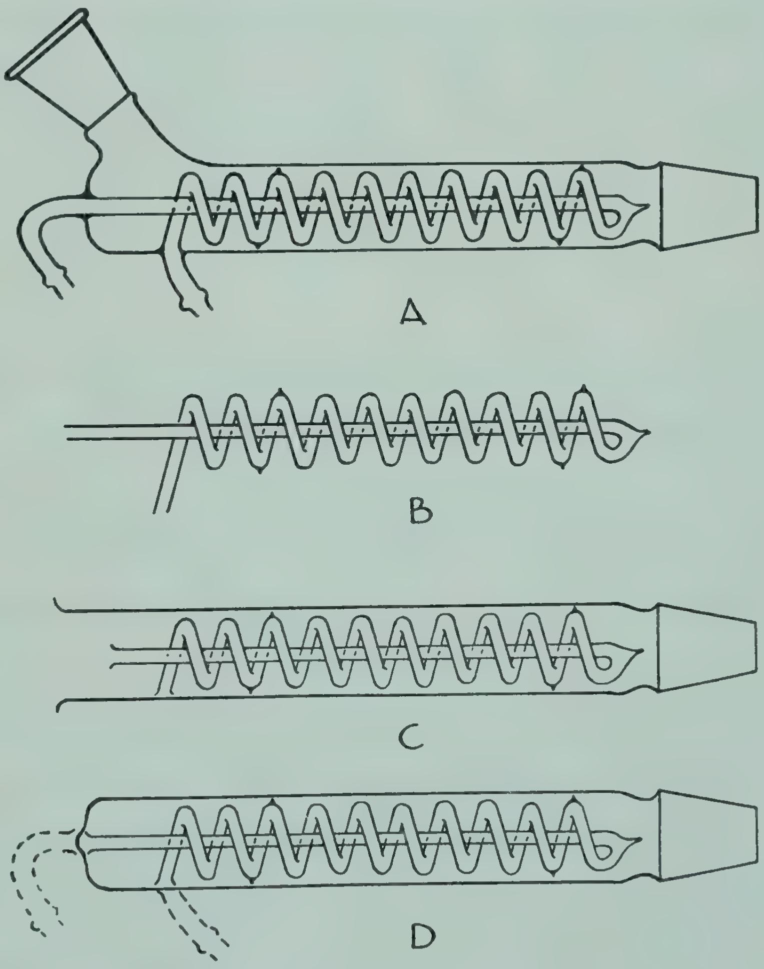

Figure 5. Reflux coil condenser.

A coil of 8 mm. O.D. tubing, which was wound on a ½ in. O.D. carbon rod, is selected and cut to the desired length. The center return tube is prepared and sealed in position as shown in

Figure 5B. Pyrex rod or a tungsten wire may be used to close the space between the two tubes if they do not meet evenly. Glass rod supports or spacers should be sealed to the coil and these should be the proper length to permit the coil to slide smoothly into the tubing selected for the jacket.

The jacket is prepared by sealing a $ inner joint to one end (Figure 5C) and the section of coil is placed in position.

During the heating and drawing off of the excess length of tube forming the jacket, the jacket must be rotated at a slight angle to prevent the coil from moving against the hot glass and sticking. When the end is rounded off, the coil is moved into position for sealing in place (Figure 5D). The end should then be heated with the flame concentrated on the area inside the circle made by the inner tube. When this is soft, it is blown out and the outside small tube is sealed in place and bent. The other outlet is sealed in place, employing the techniques described on page 97 for ring seals through side walls. It is advisable to prepare a "maria" (page 83) on both outside hose nipples before sealing the hose nipples in place.

Kjeldahl Traps

These traps give the glassworker the opportunity to make bulbs, but they also can be made from commercial flasks. A glass tube wrapped with asbestos paper to serve as a handle during the making of the first seal is convenient. Preparing the various parts for the trap is shown in Figure 6A. A small hole is blown out in the bottom of the flask of such a diameter that the small piece to be inserted fits snugly (Figure 6B). The actual seal is made by heating the glass at the juncture of the tube and flask and pushing slightly to assure even contact. When the seal is completed, it should be carefully annealed before beginning the second insertion seal (Figure 6C). With the first insertion seal acting as a handle, a second hole is blown in the flask at an angle 180° from the first. This hole should be blown to the correct size for the maria on the second insert to fit, and the seal is made with the same technique employed on the first seal. If the Kjeldahl flask is to be used for trapping out and returning liquids or mercury, as in a trap protecting a McLeod gage, a small hole should be blown just above the maria before the insertion seal is made, as shown in Figure 6D.

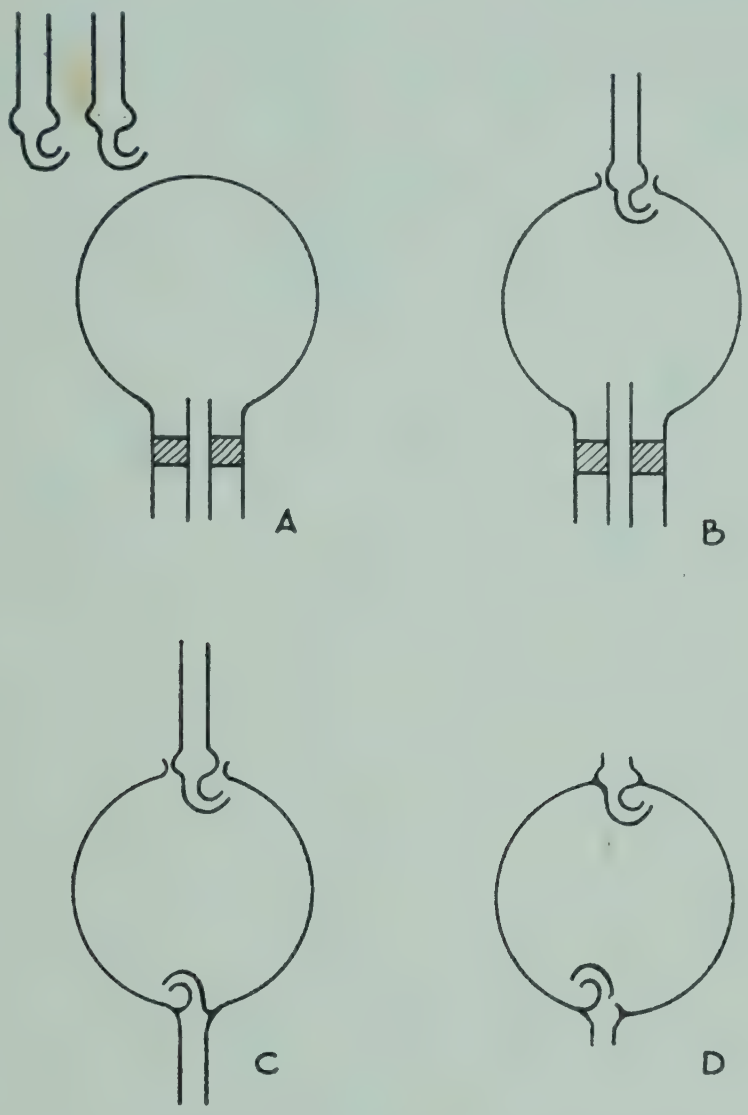

Figure 6. Steps in making a Kjeldahl flask.

When difficulty is experienced in getting the flare around the hole in the flask to shrink evenly against the maria, the seal may be started with the flask supported in a ring stand and by means of the spot-heating technique, a section of the flare is heated and pushed against the maria. This spot heating is continued until the seal is completed, at which time it is removed from the ring stand and finished by being rotated in the burners. During this heating, the seal will become smoother if the right hand maintains a slight pressure towards the left hand, and an occasional puff of air is directed into the flask to round out the area around the seal.

Gas Flowmeters

There are two types of flowmeters that can be made easily with a little practice. Figure 7 illustrates the steps for making a compact concentric-tube flowmeter. This type of flowmeter has other advantages besides compactness. It is easy to mount and can be made in several different lengths. By keeping the diameter of the inner tube small in respect to the outer, the flow can easily be ascertained by noting the difference in height of the liquid in the inner and the outer tubes. Graph paper pasted behind or to one side of the flowmeter is helpful.

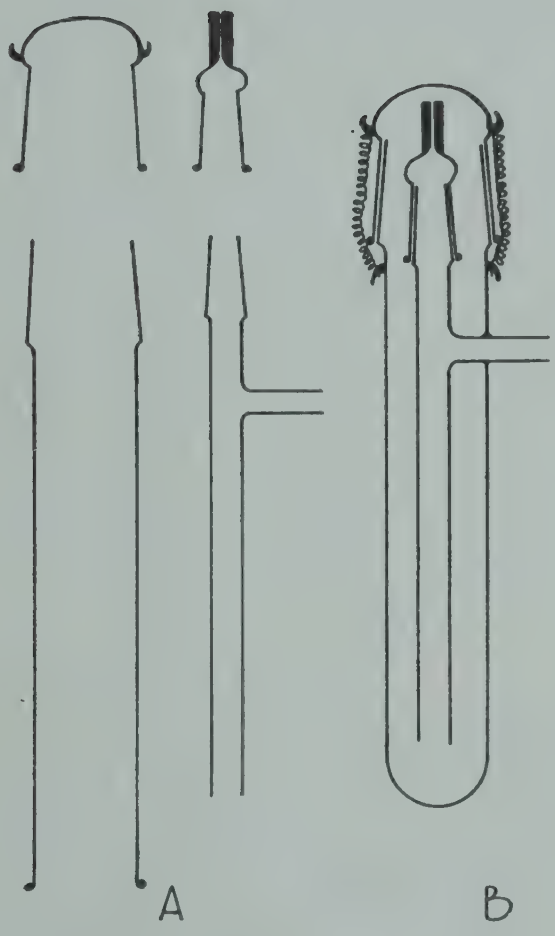

Figure 7.
Concentric tube flowmeter.

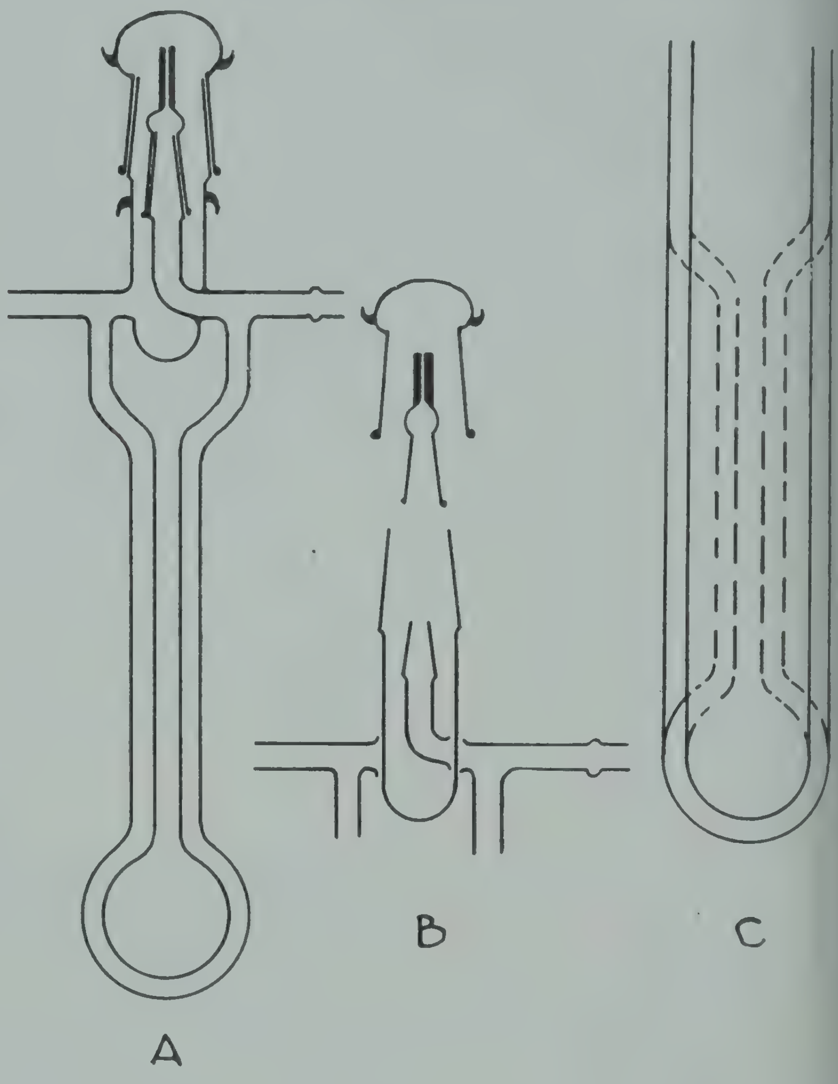

Figure 8. U-type flowmeter.

Figure 8 shows a conventional U-type flowmeter. These are made by making up the parts as shown in Figure 8B. The bend is made first in a large U shape (Figure 8C), and then the sides are heated and bent into the position shown in the dotted lines. The 10/30 ₰ joint is sealed into the 24/40 ₰ joint following the procedure given on page 97, and the bend sealed in place. This flowmeter, although more fragile than the concentric tube model, is also more accurate and easily read; therefore, this design is more desirable for highly accurate work.

Thermometer Wells in Boiling Flasks

In preparing boiling flasks, as is true with nearly all types of glassblowing, there are several ways to obtain a given result. The following series of drawings are self-explanatory and show three ways of inserting a thermometer well in flasks (Figure 9).

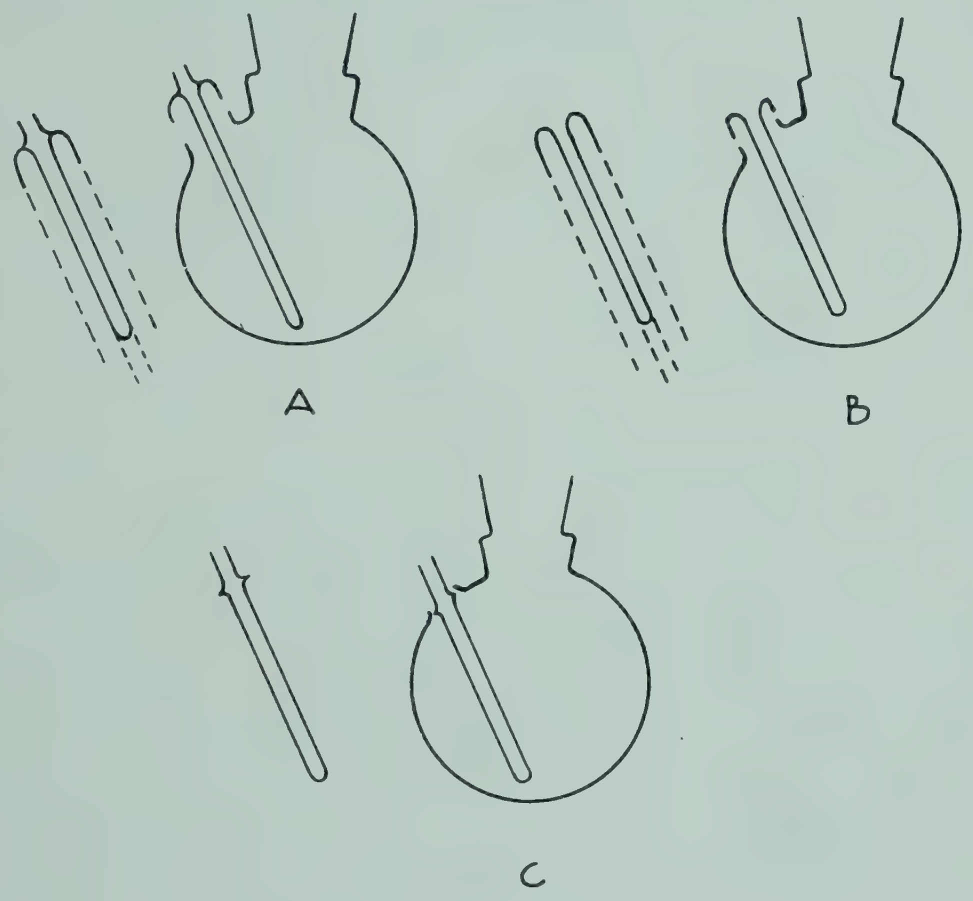

Figure 9.
Three methods of sealing thermometer wells in flasks.

Manometers

The concentric-tube type of manometer like the same type of flowmeter, is the most compact, rugged and easily made. Filling too is simplified with this type of manometer (see page 375). No support is necessary in sealing the center tube in concentric-tube manometers, since the flared end of the tube is supported in the rounded end of the outer jacket, and the small glass supports at the constricted end keep it centered. The side arm is sealed on immediately after the completion of the end seal. For some work, two side arms, as shown in Figure 10C are necessary. Figure 10A shows the construction steps for these manometers as well as the design for the conventional U-type manometer and open end manometer. See Figures 10D and 10E.

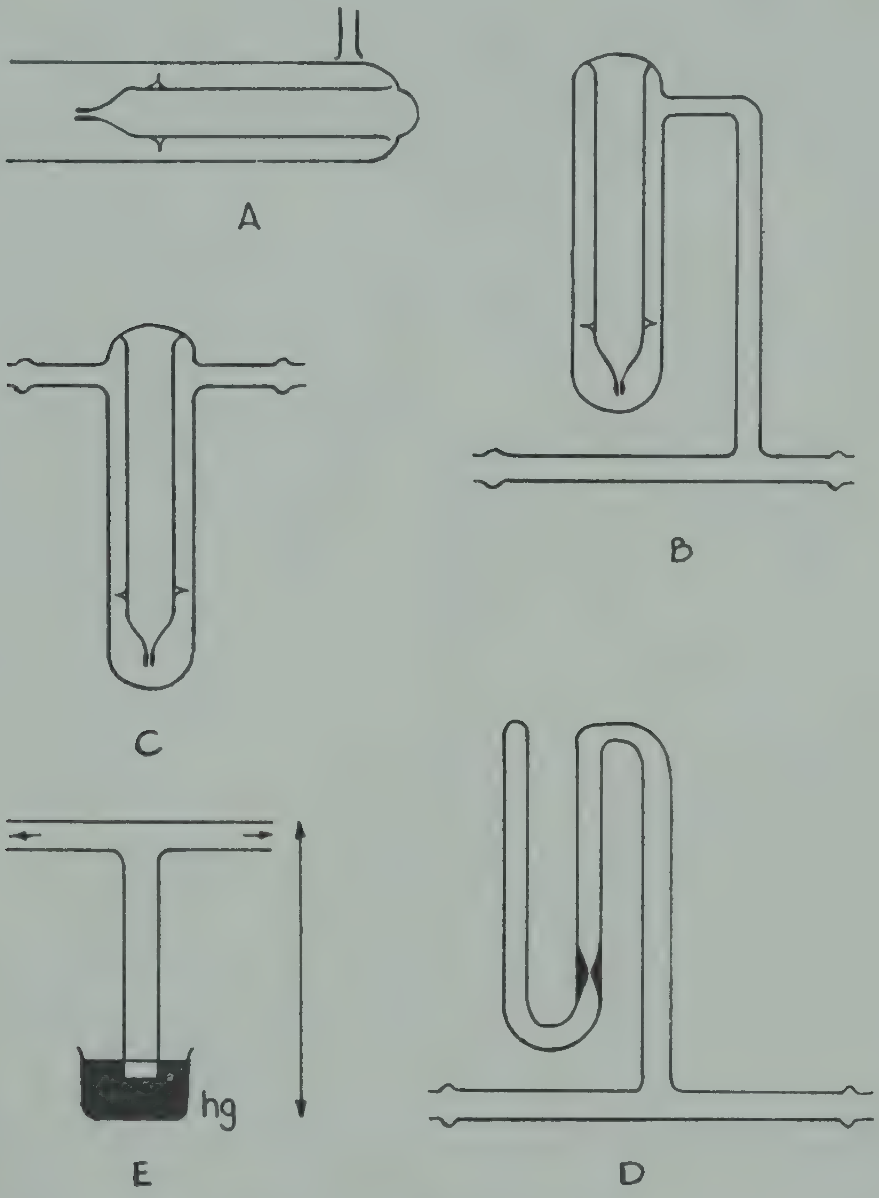

Figure 10. Manometers.

Liquid Air Traps

Dewar. Vapor traps represent a compromise between high pumping speed and efficiency of condensation. The Dewar type trap permits greater pumping speeds than the trap shown in Chapter VI, Figures 1 and 2, but it is also less efficient as a condenser. Following one of the methods described for making Dewars (page 84), the trap is assembled and made in the order shown in Figure 11. When the Dewar seal is completed and given a preliminary annealing, the side arm is sealed on. This may be either straight tubing or $ joint, depending on the intended use of the trap. With a handle supported, as shown in Figure 11C, the bottom end of the trap is finished up as desired.

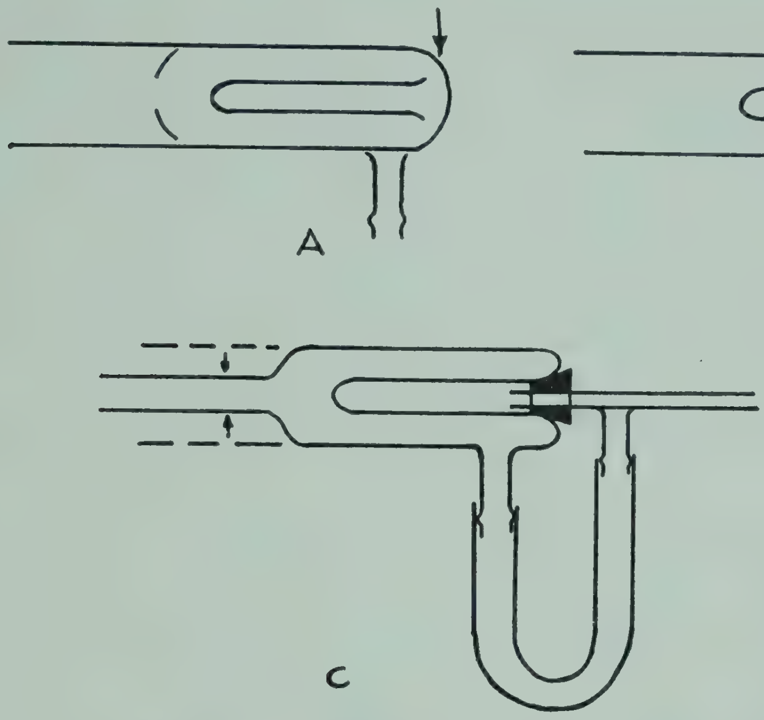

Figure 11. Liquid air Dewar traps.

Spherical Liquid Air Traps. A modification of this type of Dewar trap may be made from two sizes of boiling flasks, as shown in Figure 12. The outer flask is prepared by sealing a 10- to 12-mm. tube to the bottom and, with this as a handle, pulling the neck off flush with the periphery of the flask (Figure 12B). After the flask has been well annealed, it is allowed to cool and then cut in two with an electric hot wire (Figure 12B). The inner flask is then prepared by cutting the neck off short enough to allow the flask to be supported in the two halves of the larger flask as shown in Figure 12C. Either copper cuttings or folded copper screening may be used for supporting the inner flask in this position.

The outer flask should be set in a Transite ring or some other suitable support which will prevent it from rolling during the sealing process, and the whole assembly should be well preheated with a large (Litton) air annealing burner before the actual sealing with a gas-oxygen flame is begun. This heating with a gas-air flame should be continued during the entire sealing operation to prevent the flasks from cracking. The weight of the top half of the outer flask should be enough to assure that the glass flows together when heated. Should a space open between the two sections, it will be necessary to use small-diameter Pyrex rod to close the opening.

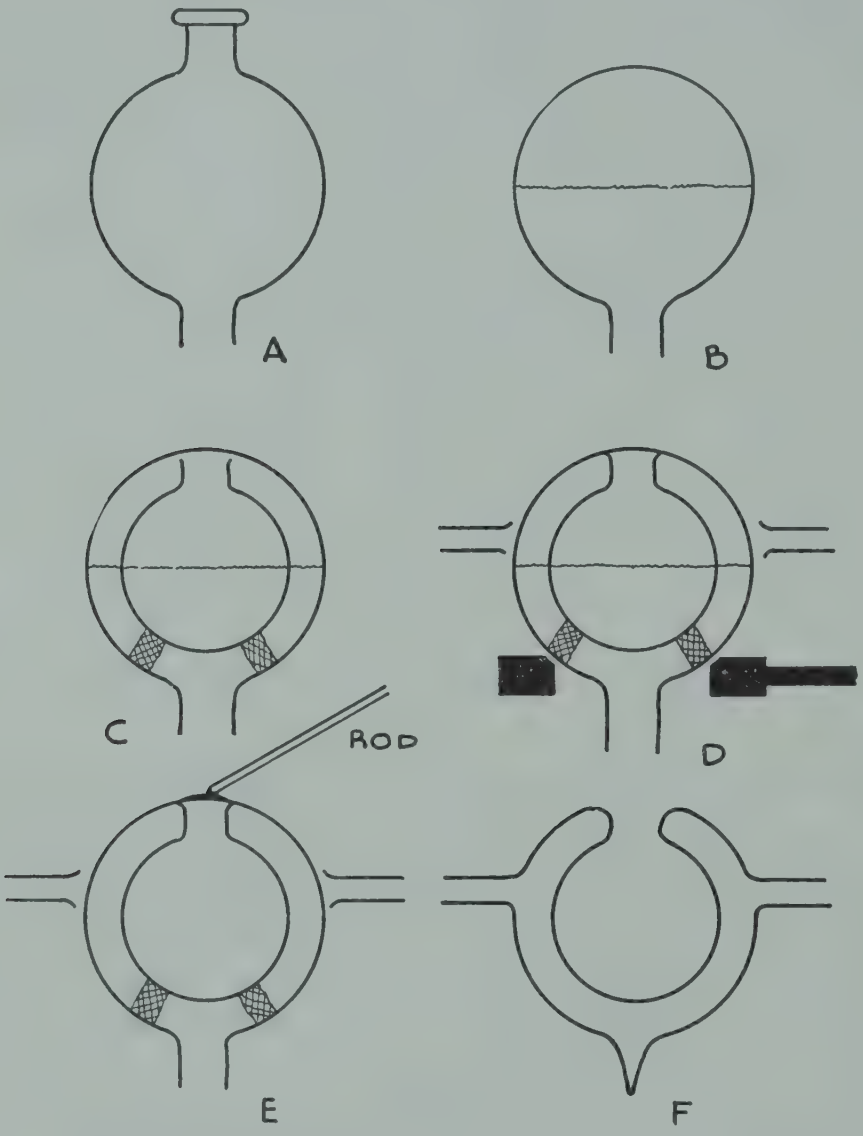

Figure 12. Spherical liquid air traps.

In making a large stationary seal with a hand torch, it should be remembered that the longer the glass is heated, the more distorted it becomes. To minimize this distortion at each spot, the glass should be blown in and out as few times as are necessary to remove the line of seal and make the glass flow evenly. At no time should the flask be blown to a larger diameter than the original flask, since this accentuates the downward flow of glass. The seal can be made almost invisible if the edges of each half of the larger flask are washed with soap and water, rinsed clear, and dried with a clean towel just prior to starting the seal, provided of course the glass is not overworked during the sealing operation.

The flask should be thoroughly flame-annealed as soon as this large seal is completed, after which the top of the outer flask is heated and joined to the neck of the inner flask. When these two flasks are firmly sealed, the hot air in the closed-off inner flask will expand and blow out through the neck. The glass at this spot can be thinned by being heated and pulled away with a glass rod until it becomes thin enough to break open (Figure 12E). The excess glass around the seal is removed by peeling as in making Dewars. The Dewar is finished up by the spot-heating method and by smoothing the seal with a carbon rod. Some practice will be necessary before professional looking Dewar seals can be made by this method. However, again it is advisable to use a small hot gas-oxygen flame for making the seal while keeping the zone of operation hot with a second or auxiliary gas-air torch. When the Dewar seal is completed, two side arms should be sealed on and the bottom tube tipped off as shown in Figure 12F. The bottom tube is useful for opening and removing liquids should some condense out in the trap.

Double Condenser

This type of a condenser consists of a cold finger condenser within a straight water jacket (see Figure 13A.) To make these condensers four different sizes of tubing are chosen, starting with the smallest tube and working out. The choice of sizes should provide a condenser with sufficient free space inside to handle the amount of condensable material expected as well as enough clearance between the two surfaces of the water jacket to make strong ring seals. The jacket for the cold finger should be prepared first (Figure 13B), with a small hole left in the tip for blowing through.

The inside water inlet tube of the cold finger is held in position during construction by two sets of glass spacers sealed toward each end (Figure 13C). These should be the proper length to give a smooth sliding fit in the jacket. The cold finger and the inner tube of the straight water jacket are supported with copper screening (see Figure 13D).

During the actual glassblowing operation, ring seal 1 is made between the inner and outer members of the water jacket. The extension tube sealed in place should be as large as or slightly larger than the inside tube of the water jacket. When this ring seal is complete the hose connection for the water jacket is sealed in place. It is important that both the ring seal and the end be kept hot by intermittent heating over a Bunsen burner during the entire process of making three ring seals and sealing on the separate side arms. When the first ring seal and side arm are finished the cold finger is pushed forward in position as shown in

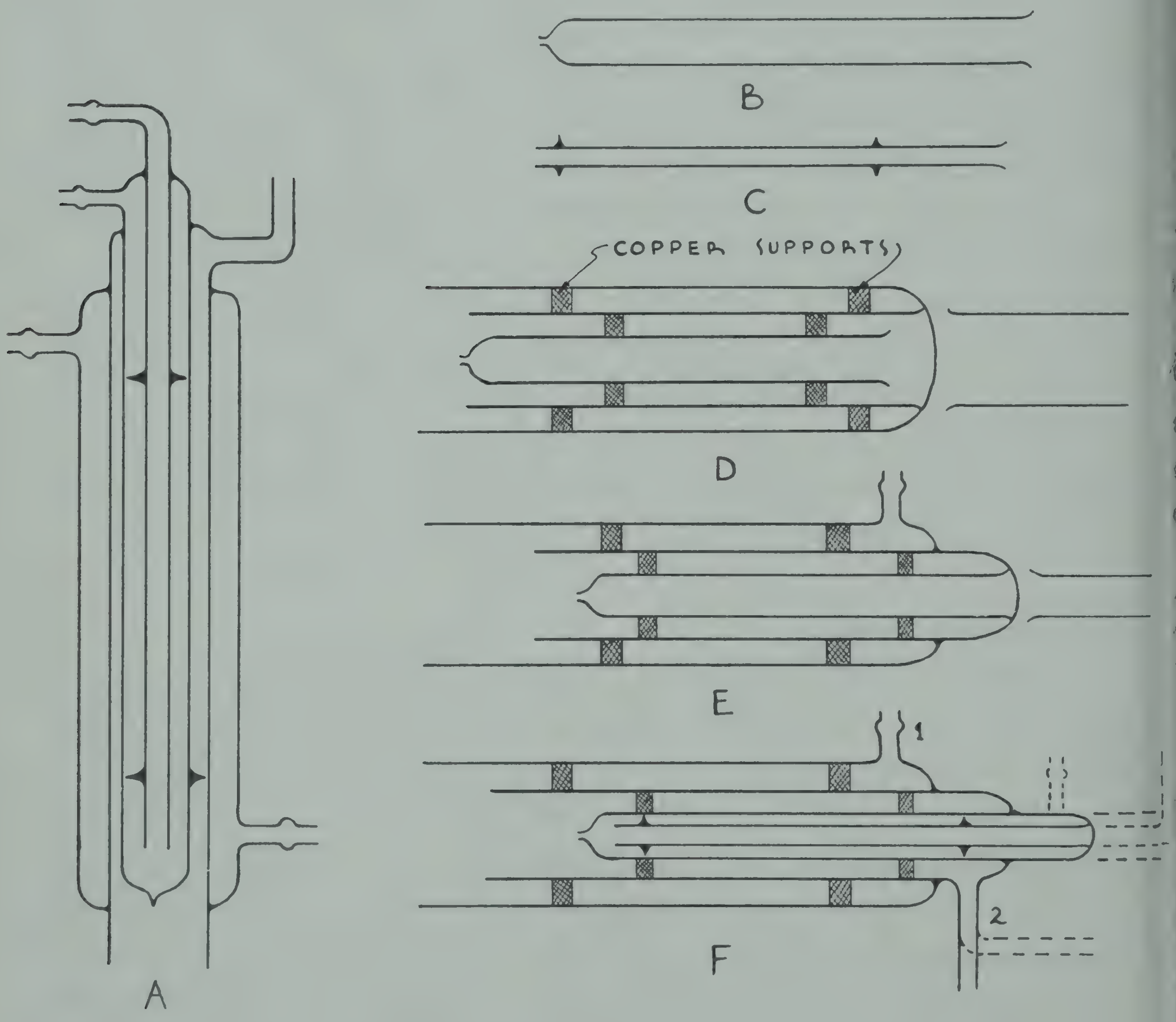

Figure 13. Double condenser.

Figure 13E and the second ring seal is made. The side arm forming a vent to the inside of the condenser is then sealed in position. The completed work is briefly annealed, after which the smallest tube is placed in position, the final ring seal made, and the water outlet tube for the cold finger sealed in place. The entire triple seal should be thoroughly annealed before allowing the glass to cool to room temperature. If the water outlet (3) is left straight, it makes an ideal handle for attaching the blow tube and rotating the piece during fabrication of the ring seal at the opposite end. With hose nipples 1 and 2 connected by a short piece of rubber tubing, blowing is even, both in the water jacket and within the condenser. After the ring seal is formed and the end blown out just prior to sealing the extending piece of tubing in place, the hole in the bottom of the cold finger can be closed by heating with a hand torch and alternately blowing and sucking. After the last two steps of completing the bottom ring seal and sealing the water inlet tube in position, this end is also well annealed and allowed to cool. Cold finger outlet (3) can then be bent to the desired position; care should be exercised to avoid playing a flame directly on any of the top ring seals.

Spherical Condensers

A spherical condenser is made by sealing three flasks, one inside the other. When compactness with a large cooling surface is desired, spherical condensers are excellent. (See Figure 14.) As in the construction of the spherical liquid air trap, these condensers are made by cutting flask No. 2 in half with a hot wire for inserting the inside flask (No. 1).

Flask 1 is prepared as follows. A tube of 15 to 16 mm. O.D. tubing is cut, flared, and inserted in flask 1 as shown in Figure 14A. It can be held in position with asbestos paper and when the ring seal is finished, the water inlet and outlet tubes are sealed in place (Figure 14B). It is important that these tubes be sealed close to the ring seal to facilitate sealing flask 2 in position. When this end has been completed, annealed, and allowed to cool, the Dewar-type seal on the other end is made by sealing off the neck of the flask, reheating the bottom and drawing it in until contact is made with the inner tube. The glass inside the circle formed by the inner tube is blown out and the Dewar completed by paddling the glass into shape with a carbon rod, after which three or four glass supports are sealed in place (see Figure 14C).

Flask 2 is prepared by cutting it in two with a hot wire and cutting or grinding the neck off to form an opening large enough for the top of flask 1 to fit through (Figure 14D). The opening around the neck and the inlet and outlet tube can be filled with shredded asbestos paper which will form an airtight seal for blowing while flask 2 is sealed together and the glass supports sealed between Flask 1 to flask 2. This operation can be done with a hand torch while the flask is supported in a ring stand. A second gas-air annealing burner should be used to keep the glass hot and prevent cracking. If the condenser is to be used for condensing

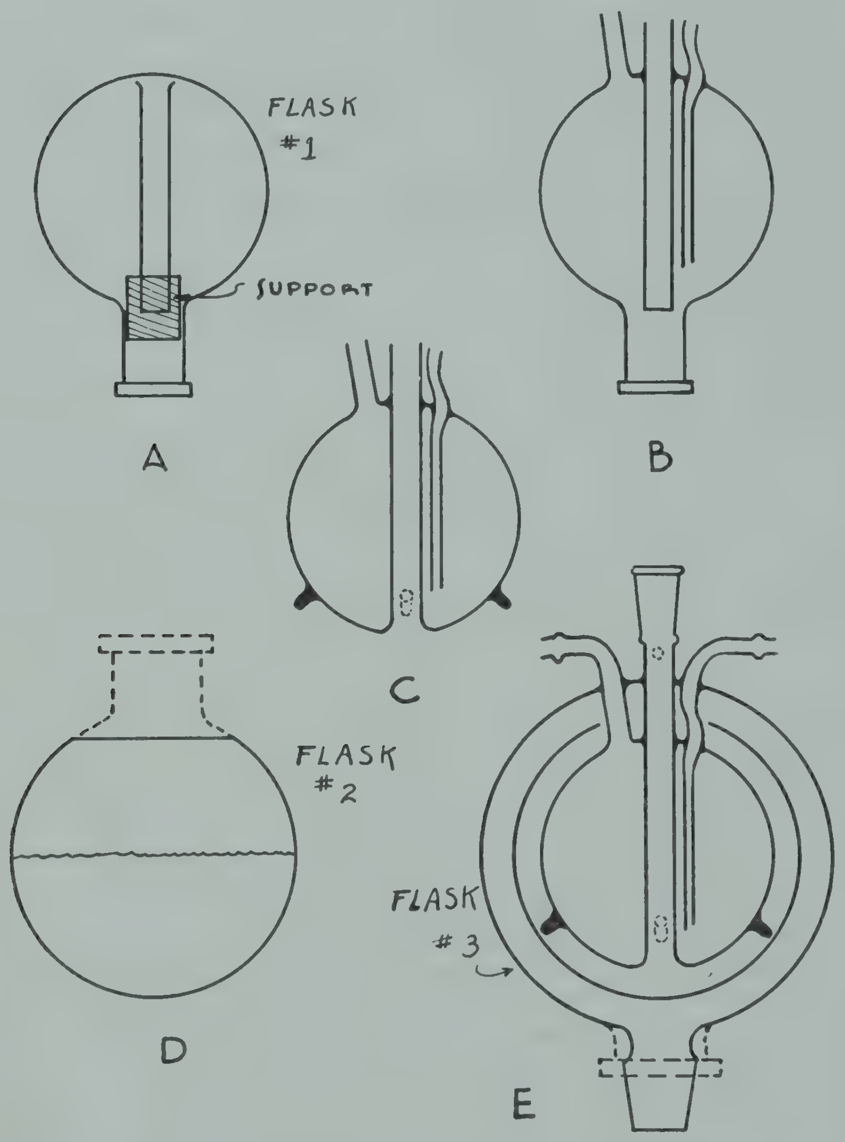

Figure 14. Spherical condensers.

considerable amounts of liquid, the bottom of flask 2 should be prepared with several holes for returning the condensate, with the number of holes kept to a minimum so that the condensate will form a liquid seal and prevent vapors from escaping through the holes. The outer, or flask 3, can be prepared by cutting it in two and resealing it around the other two, or it can be prepared by flaring the neck wide enough to admit the other two flasks, which are then sealed in place and the neck drawn back down again to form the bottom.

In place of a third flask, a piece of standard-wall Pyrex tubing of the proper diameter may be used. Such a tube is prepared by making a closed test-tube end through which the top and water lines of the inner flask are sealed, after which the opposite end is rounded off and the $ joint sealed in position. The appearance of a condenser made with glass tubing will be improved if the curvature of the drawn-off ends is approximately the same as the curvature of the flasks inside.

Inverted Ring Seals

Three different sizes of tubing are selected for making inverted ring seals, beginning with the smaller or inner tube and working out. In making the seal, the inner tube is ring-sealed through the second tube according to the usual procedure for ring seals. The seal is annealed and allowed to cool, after which the outside tube is cut to the desired length of inversion by means of the electrical hot wire. The resulting end, if jagged, is made smooth by careful

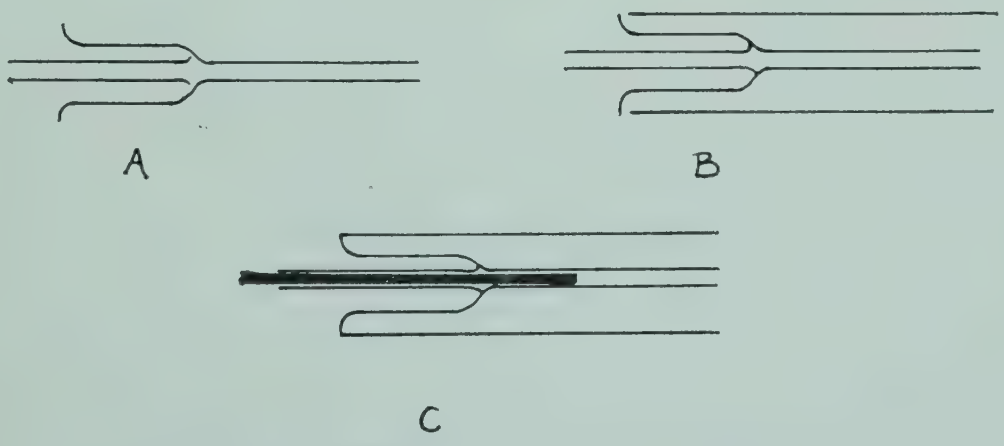

Figure 15. Inverted ring seals.

peeling and spun to a slight flare with a triangular flaring tool or small carbon rod. This flare is finally sealed to the third or final outer tube. Deformation of the small center tube during this heating is minimized by the insertion of a small carbon rod in the center tube. (See Figure 15C.) If a side arm on the inverted seal is necessary, it should be added at the time the seal is originally made.

Glass Turbines

When it is necessary to circulate gases in a closed system, the glass turbine designed by Beeck[1], shown in Figure 16, may be used. If a glassblowing lathe is available, making the shell for the turbine is simplified; however, equally efficient turbines may be made by hand. With practice it is possible to draw the three diameters of tubing from the larger size which houses the paddles by

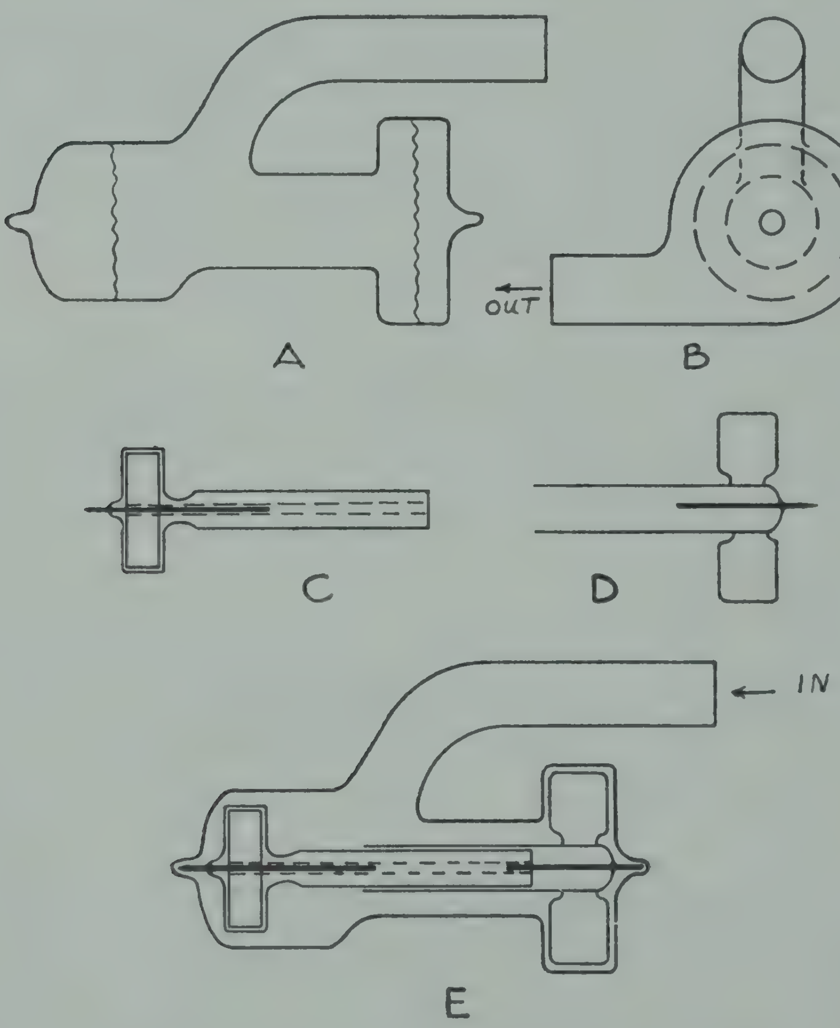

Figure 16. Glass circulating turbines.

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[1] Beeck, O., *Proc. Roy. Soc.* (*London*), **A177**, 65 (1940).

first drawing down a section to the smaller diameter and later blowing this back to the diameter required to enclose the soft iron rod. The shell may also be made by sealing tubings of three different diameters to the correct length.

The inner shaft is made in two sections. The part to which the paddles are sealed is made from standard-wall tubing (Figure 16D), and the soft iron core is sealed to a section of capillary tubing (Figure 16C). The capillary tubing chosen should have the same I.D. as the O.D. of the tungsten used for the bearing, and the capillary may be used for holding the tungsten bearings centered while they are being sealed in position. The protruding end of tungsten on each piece of the inner shaft should be brought to a sharp centered point by being heated to a red heat, and rotated immersed in sodium nitrite. In assembling the turbine, both ends of the shell are cut off with a hot wire as shown in Figure 16A, and the inside parts assembled in position. The end of the shell over the soft iron core is held in place by cellulose tape while the seal is made in the section housing the paddles. Considerable care must be exercised to prevent the shell from touching and sticking to the paddles, since the clearance between the paddles and the shell should be kept at a minimum to produce an efficient turbine. When the seal in the paddles section of the shell is complete, the outlet arm shown in Figure 16B is sealed in place and this end of the turbine annealed. The cellulose tape holding the opposite end, covering the iron core, is then taken off, the shell cap removed, and the two pieces of shaft adjusted to the proper length so that both tungsten points seat in the glass bearings. This adjustment is made easily and accurately if section C is left extending from section D, making the shaft too long. The cap end of the shell should then be carefully set in place, thereby pushing the two pieces of the shaft to the proper length. The outer shell piece is then removed and the shaft sealed together. This should be done quickly and without excessive heating in order to prevent the standard-wall tubing from blowing out from the expanding air inside, after the seal is completed. The second seal is made in the outer shell, after which the inlet tube (Figure 16E) is sealed in place.

The turbine should be oven-annealed if possible, and the final adjustment of the inner shaft made later. Final adjustment consists of heating the motor end (iron core) and pressing the glass in to take up any looseness in the bearing shaft. The soft iron core, and thus the inner shaft, will be more evenly balanced if a hole is accurately centered and drilled through it to allow the tungsten bearing to pass through.

Glass turbines may be powered either by revolving magnets attached to a motor, or by induction current, turned on after the turbine is started with revolving magnets.

Glass Expansion Bellows

Expansion bellows are used to compensate for differences in temperature between two pieces of rigidly sealed tubing. One of the more common uses for expansion bellows is on the vacuum jackets of fractional distillation columns.

Expansion Bellows Made by Hand. Bellows may be made by hand by thinning the glass and blowing it into a doughnut shape (see Figure 17A). The bellow is more sensitive if, after the doughnut shape is blown, it is quickly pushed together, and a final puff of air directed into it before the glass sets, to give a shape like that shown in Figure 17B. The objection to this type of bellows is that the thin glass is outside of the tube, where it is more easily broken. As a general rule, this type of bellows can be made only by an experienced glassblower.

Expansion Bellows Made on a Lathe. Pompeo and Meyers[2] have overcome these objections by making bellows on a glass lathe and placing the thin glass inside the tube, where it cannot be broken so easily. Making glass bellows on a lathe is done in four separate steps and when these are followed carefully, the bellows will have a predictable behavior.

Step No. 1. A 10 mm. section of glass tubing is heated while being rotated in a glassblowing lathe and, when soft, the glass is drawn by moving the tailstock so that the heated section is stretched about 12 mm. (See Figure 17C.) The location of the burner and the flame adjustment are important for the successful production of uniform and flexible glass bellows. The desired stretch can best be obtained if only the small length of the original tube is heated prior to being stretched. By setting the burner close to the glass (about ½ to ¾ in.) and adjusting the flame to small, intensely hot tips with the minimum of splash, heating can be confined to a width of about 10 mm.

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[2] Pompeo, D. J., and Meyers, Edward, *Rev. Sci. Instr.* **12,** 368 **(1941)**.

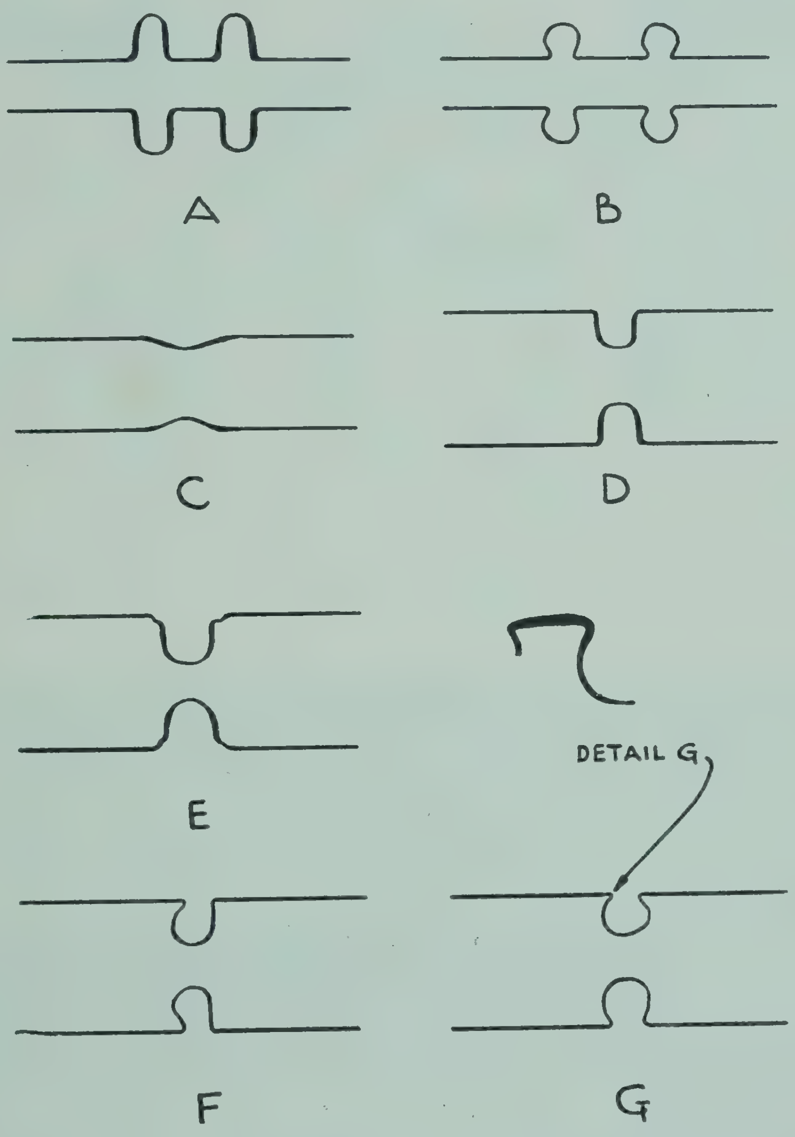

Figure 17A-G. Glass expansion bellows.

Step No. 2. With a small hot flame, the thin section produced in step 1 is reheated and depressed about 10 mm., by means of a 1-cm. carbon rod held at right angles to the glass. The results of this operation are shown in Figure 17D. If too wide a section of tubing was heated in step 1, step 2 will produce the results shown in Figure 17E. This will probably result in bellows poor in appearance and unpredictable in performance. These should be discarded.

Step No. 3. By means of a gas-oxygen hand torch, the glass is heated at the shoulder and the tail piece moved back about 5 mm. to produce the shape shown in Figure 17F. When bellows are being formed in very large tubing, heating the shoulders with only a hand torch may be slow. This operation may be speeded up by setting the lathe burner just back of the shoulder and adjusting the flame to allow only the tip of the gas-oxygen flame to touch the glass.

Step No. 4. The other shoulder is heated with a hand torch and pushed in about 5 mm. leaving an opening of about 3 to 4 mm. (See Figure 17G.) As many bellows may be formed as is desired by repeating these four steps, leaving 1.5 to 2.0 cm. of standard-wall tubing between each convolution. Glass bellows should be thoroughly annealed before being removed from the lathe. Meter sticks mounted on the lathe are very helpful in the various steps of making glass bellows, especially when the burner and tailstock are fitted with the pointers also shown in Figure 17H.

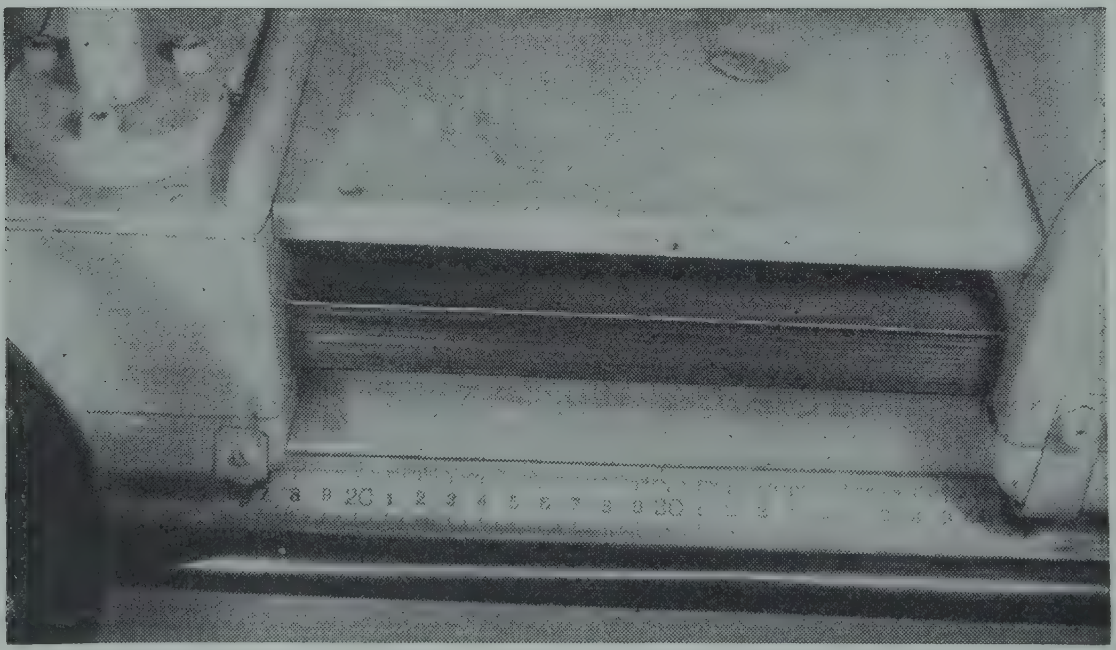

Figure 17H. Glassblowing lathe adapted for making glass bellows.

In most cases where expansion bellows are built into glass apparatus, it will be possible to prepare the bellows ahead of time and assemble the apparatus in the usual fashion. In special cases it is recommended that the apparatus be assembled as shown in

Figure 17I, the bellows formed, and then the final seals made. This can usually be done without damage or distortion to the inner tube, since only a small area of the outside tube is heated during the formation of the bellows and the heat is applied for only a short time.

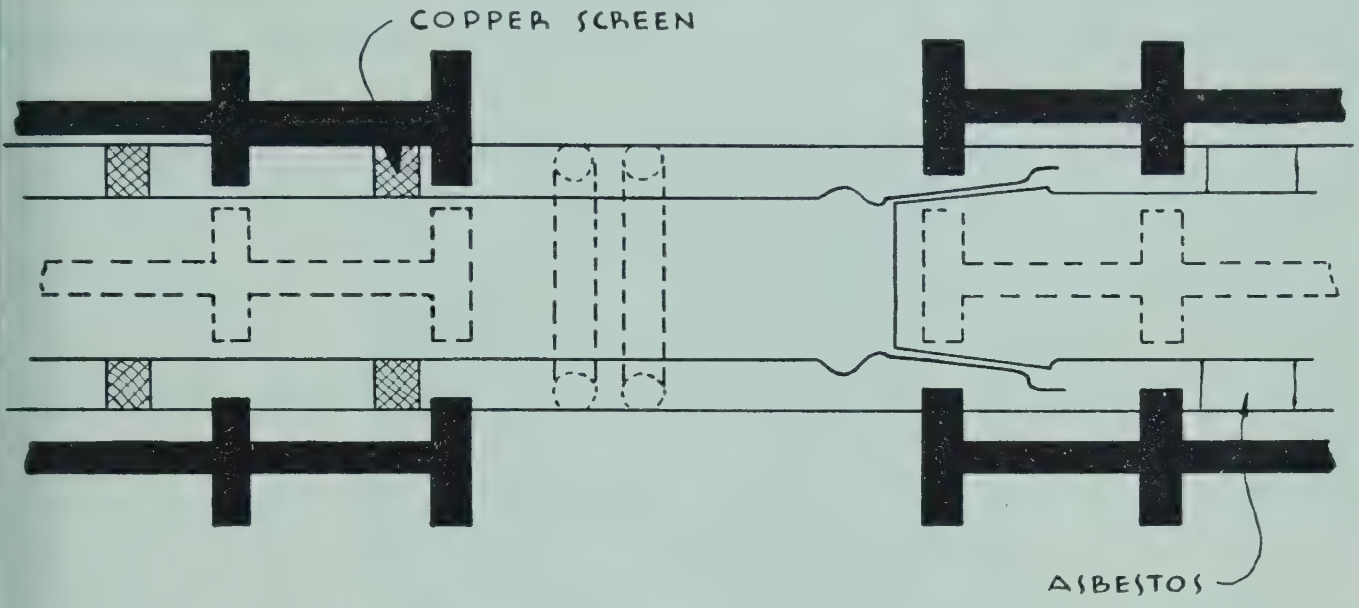

Figure 17I. Glass expansion bellows made on a lathe.

Specifications for Glass Expansion Bellows. A thickness of 0.4 to 0.5 mm. at the bottom of the convolution, gradually increasing to 0.8 to 0.9 mm. at the top or shoulder, will produce bellows of good flexibility. Practice bellows should be made and tested until bellows can be reproduced consistently which will not only be flexible but also be able to withstand a tensile force of at least 35 pounds.

According to Pompeo and Meyers, a 117 cm. column containing seven convolutions, had an overall expansion of 0.48 mm. when the inside operating temperature was 191° C. The contraction of the same column was 0.46 mm. when the inside operating temperature was —160° C. This corresponded to 2.4×10^{-6} and 2.2×10^{-6} (assuming the jacket temperature was 20° C. in both cases), which is considerably less than the figure of 3.2×10^{-6} for Pyrex glass given by the Corning Glass Works. From these figures Pompeo and Meyers assumed that for low-temperature work a column 117 cm. long could be operated safely with seven convolutions, but for a temperature differential of 250° C. between the inside and outside jacket, the bellows should contain eight or ten convolutions.

Testing Glass Expansion Bellows. Figure 17J shows an elaborate device designed and built by Schuster[3] for testing the flexibility and strength of finished glass bellows, but a simple device may be made by clamping the tube containing the bellows rigidly in a ring stand. A metal cap or cover to which a long chain is attached is set on top of the glass tube with the chain hanging down through the center of the tube. A gage for indicating motion should be clamped touching the cap and set at zero. The actual test is made by hanging weights to the chain just below the glass tube. It is advisable to set a pan or bucket directly below the glass tube in order to catch the glass in case the bellows break.

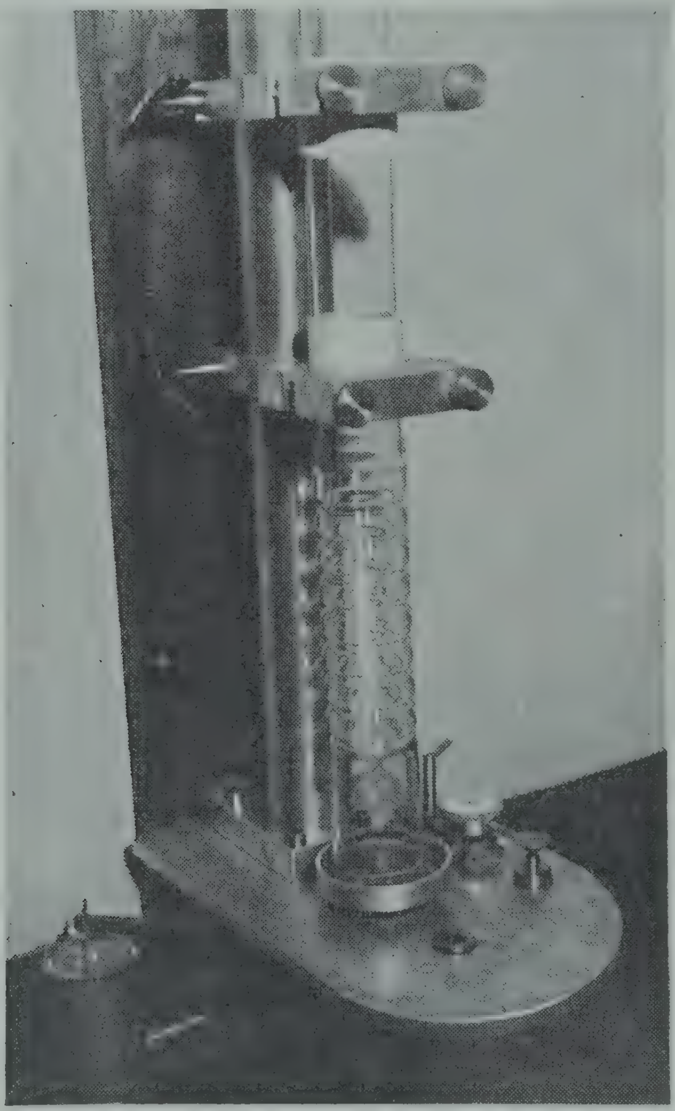

Figure 17J. Machine for testing glass expansion bellows.

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[3] Schuster, F., Chemistry Department, University of California, Los Angeles.

Satisfactory bellows should withstand a 0.10 mm. per bellow compression strain and a test load of not over 50 pounds maximum without breaking.

Distillation Receivers

A water-jacketed distilling receiver or fraction cutter is shown in Figure 18. Three stopcocks adapt this fraction cutter for reduced-pressure distillations since the receiver may be sealed off from the column, the vacuum broken, the receiver replaced and re-evacuated, all without disturbing the vacuum in the column. A calibrated reservoir, made from a 15-ml. calibrated centrifuge tube (Corning No. 8080 or 8100), is used for storing the distillate while the receiver is being replaced. The equalizing line of the vacuum manifold enters through the water jacket, reducing the possibility of the distillate's being drawn into the vacuum pump.

The following steps, shown in Figure 18B through 18F, are recommended for the construction of these cutters. The small end of the centrifuge tube is blown out and a piece of 8 mm. O.D. tubing sealed to it. With this extension as a handle, a piece of 17 mm. O.D. tubing is sealed to the large end and the constriction made as shown in Figure 18C. The constriction should be about 1 in. above the top calibration mark. About 1 in. above the constriction, the tube is closed off and a small hole blown preparatory to making an insertion seal.

The inner drip tube is prepared by forming a maria on a piece of 7 to 8 mm. tubing and cutting the tubing at an angle to form a drip (Figure 18C). The length of this tube is such that the tip will be slightly above the top calibration mark on the centrifuge tube when the insertion seal is completed. A small vent hole should be made in the drip about ¾ths of the way up the tube from the point where the angle was cut. This vent helps prevent viscous liquids from sticking in and sealing off the drip tube.

No support is necessary in making the insertion seal, since the constriction will help support the inner tube, and final alignment may be made by rotating the seal until the glass sets. When the insertion seal is completed as shown in Figure 18D, the side

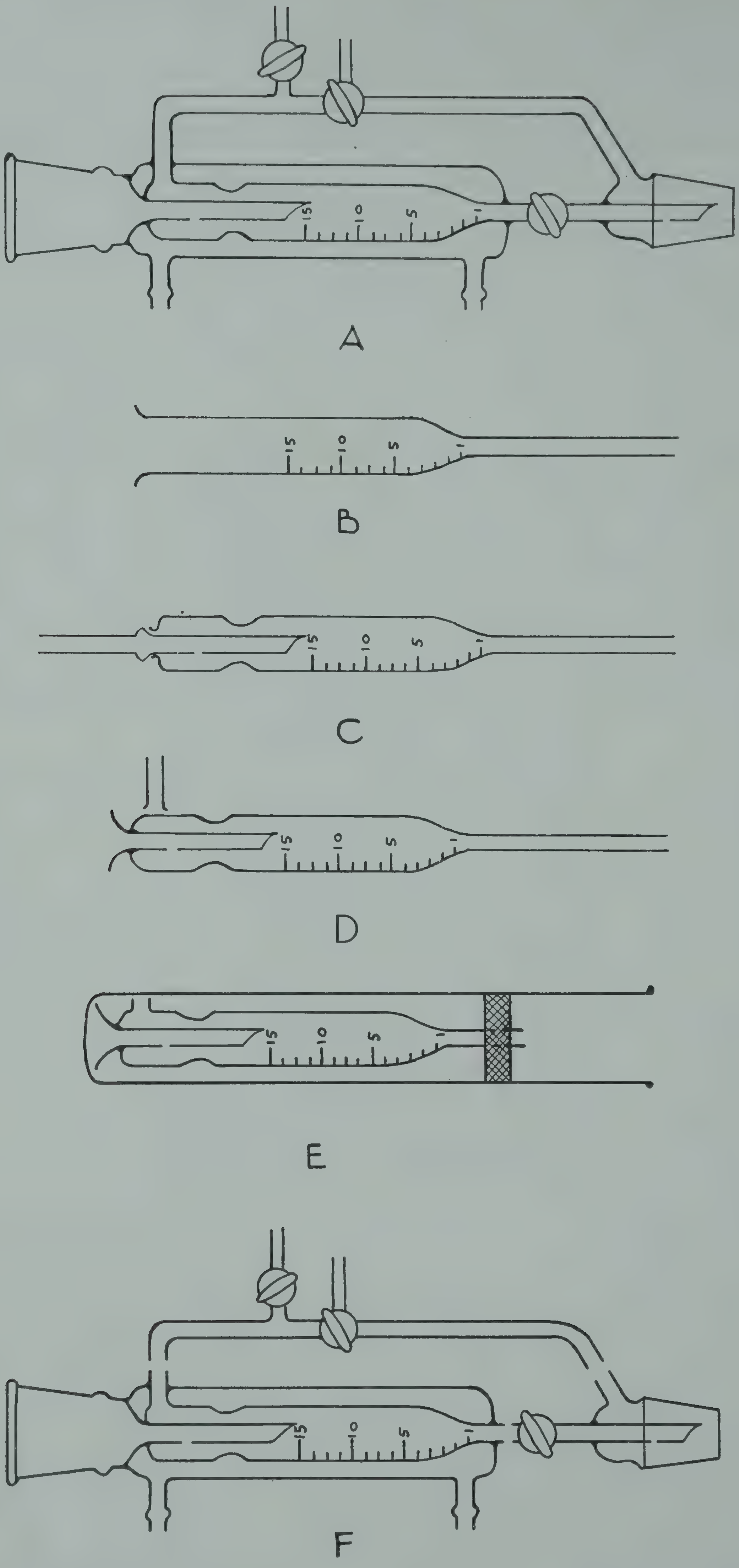
15
10
5
1
A
B
C
D
E
F

Figure 18. Steps for making a distilling receiver.

arm is attached. This assembly should be well annealed before being allowed to cool.

The water jacket is prepared from a piece of 35-mm. tubing by closing off one end as previously described in making ring seals. One support of asbestos paper is sufficient to hold the centrifuge tube in position with the flare firmly in position against the rounded end of the outside jacket (see Figure 18E). When in this position, a hand torch may be used to seal the outside jacket to the side arm of the inner section. This seal is not completed but is made merely to hold the center tube in position while the top ring seal is made and the $ 24/40 joint is attached. The final steps in the completion of the upper section of the fraction cutter are to finish the ring seal involving the side or equalizing arm (already begun) which goes through the water jacket, and to seal the water outlet to the back of the jacket.

The ring seal at the opposite end of the water jacket may be made with a $ joint handle as an aid in rotation, and a short length of rubber tubing connecting the equalizing arm and the water outlet. With this in place, blowing is equal both in the inside and in the jacket of the condenser and a straight piece of tubing may be sealed in place following the standard procedure for making a ring seal. When this is completed the second water connection (inlet) is sealed in place.

The bottom section is prepared by closing off a 24/40 $ joint about $1\frac{1}{2}$ in. above the groundsurface and sealing a piece of 8 to 9 mm. O.D. tubing through it as shown in Figure 18F. The stopcock should be ring-sealed to this section and the side arm sealed in place. The fraction cutter is completed by sealing the lower section to the main body of the fraction cutter, taking care to see that both outlets for the vacuum manifold are in line. After this is completed, and the seal annealed and allowed to cool, the vacuum manifold is sealed in place.

Addition Funnels

Addition funnels are very useful items in laboratories, especially when they are equipped with an equalizing line and a vacuum take-off (see Figure 19). Since funnels like this are not standard commercial items, they must be made. When cylindrical funnels are available (Corning No. 6380), time will be saved by starting with these. If the complete funnel is to be made, the individual parts are made and assembled as shown in Figure 19A. The finished funnel is shown in Figure 19B.

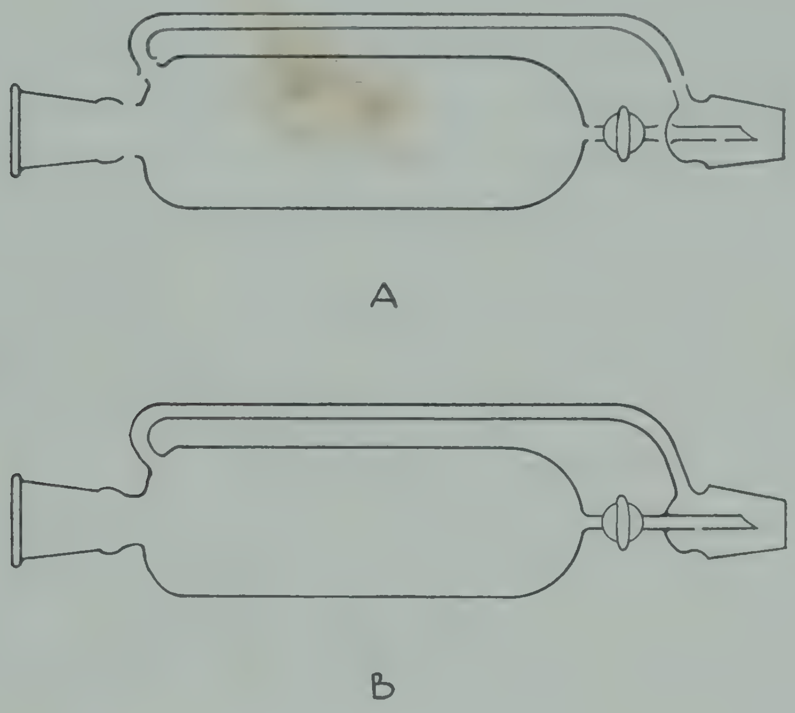

Figure 19. Addition funnel.

Vacuum Jacketed Transfer Tubes

Vacuum jacketed tubes for transferring liquid nitrogen from one vessel to another are quite easily made when the transfer tube is to be straight. A tube with one right-angle bend, however, is more difficult and a tube with two right-angle bends considerably more difficult. This is especially true if the bends are made freehand.

Transfer tubes are generally made from 12 to 14 mm. O.D. tubing for the jacket and 7 to 8 mm. O.D. tubing for the liquid air to pass through. The recommended procedure for making these tubes is to complete the vacuum jacket before making any bends. In this way the jacket is made following the procedure for making a standard condenser except that, in place of a hose nipple near the ring seal, a constricted seal-off tube is sealed to the jacket as shown in Figure 20.

Freehand bends of the two concentric Pyrex tubes may be made, since the amount of heat necessary to soften the larger tube sufficiently for bending will also soften the smaller inner tube. To avoid or minimize distortion in the inner tube, however, heating should not be prolonged and the bend should be made as soon as the glass is sufficiently soft. At this point the inner tube will bend but it is not fluid enough to cause sagging.

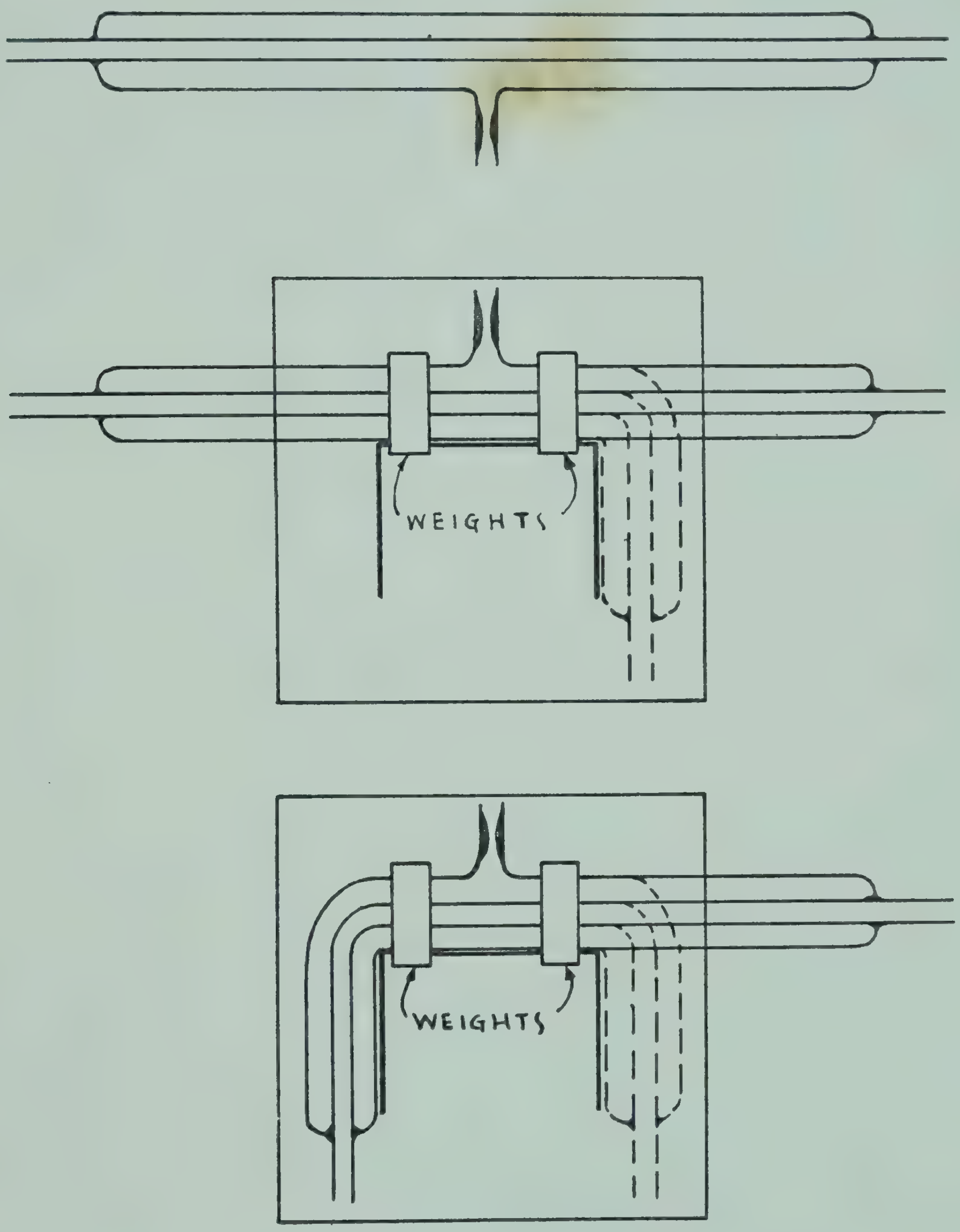

Figure 20. Vacuum jacketed transfer tubes.

When the transfer tubes required call for a vacuum-jacketed tube with two right-angle bends forming parallel legs which are centered within ±2 mm. of a given distance, freehand bending will not have sufficient accuracy. In this case the transfer tube may be heated and bent while lying on a flat piece of Transite on which a drawing has been made showing the required parallel centers. The desired length of each leg is measured and marked and a gas-oxygen flame is directed at this point to the two sides of the glass which will form the inside and outside of the curve. The bending operation is begun as soon as the glass is sufficiently soft and the combined heating and bending is continued until the desired angle has been formed. The second bend may be made by repeating this procedure. It will probably be found advantageous to turn the transfer tube over so that the second bend is made in the same direction as the first. Two weights should be placed one on each side of seal-off tube to prevent the tube from moving out of line during the bending operation.

Notes on Glassblowing Lathe Operations

With the widespread use of glassblowing lathes in the field of scientific glassblowing, no book on this subject would be complete without some mention of working procedures for lathes.

Lathe Chuck. Chucks such as the Litton Universal chucks will hold glass more firmly if small rings of either soft metal or Transite are attached to the jaws. It is also advantageous to have the back set of rings movable so they may be adapted to various lengths of tubing. See dark sections, Figure 21.

Flasks too may be held more firmly and concentric by means of these jaw rings, which should be adjusted to the size of the flask. Glass should be protected by at least one layer of asbestos tape to prevent its being cracked by too tight a grip of the jaws.

Chuck Speeds. Most lathes have some method for varying the speed at which the chuck rotates. The ability to vary this speed is important and the choice of speeds used will depend on the type of work being done. For general sealing work the glass should be rotated at a speed which will maintain the diameter of the glass tubing constant when it is heated. Too rapid a rotation will cause the glass to expand or flare, whereas too slow a speed will cause the glass to thicken and shrink when it is heated.

Chucking Glass Tubing. Centering glass tubing to turn concentrically in the lathe is often difficult because of the inherent bow in the tubing. For this reason it is advisable to plan the work so that if possible, it may be completed before being removed from the lathe. Some adjustment of the centering is possible by means of asbestos tape shims placed between the jaws of the chuck. In extreme cases it will be necessary to center the tubing by heating a section of tubing near the jaws and trueing only the end of the tubing.

Achieving true centering of the tubing by means of shims is a trial-and-error process; however, the time spent is not wasted since both the speed and ease with which the work is done and the accuracy of the finished product will be improved by having the tubing run true.

Cutting Glass Tubing in a Lathe. Cutting glass tubing in a lathe is done by means of the fire cutoff method. The burner is raised to within ½ to 1 in. of the glass and adjusted to give a small hot flame with the minimum of splash. With this flame adjustment the glass is rotated and stretched by moving the tailstock away from the lathe head. The burner should also be moved slightly in the same direction as the tailstock, thus keeping the flame at the edge of the thinned section. Air shoud be directed inside the tubing in light puffs to maintain the tube at a constant diameter while the glass is being thinned. When the glass is extremely thin the tailstock should be moved rapidly to the right while at the same time a sharp puff of air is directed inside the glass. This will result in the glass's separating evenly. The edge of the piece of tubing to be used may then be heated, thickened, and shaped to the desired form.

Drawing down Tubing Diameter. The speed of rotation of the lathe during the drawing-down operation should be slightly slower than that used for regular sealing operations. The glass should be held in the chuck and rotated while at the same time a gas-oxygen flame is directed at the spot where the reduction in size is desired; continued heating under these conditions will cause the glass to thicken and shrink. At this point the tailstock should be moved away from the lathe head (to the right) to draw out the glass and maintain an even wall thickness. The speed with which the drawing-down operation is performed is increased if a carbon rod is used to help form the glass. The concentricity of the smaller diameter section in relation to the original large piece of tubing is also improved by the use of a carbon rod.

A tubing of very large diameter would become too thin if the drawing down just described were continued until a closed end was formed. To avoid this thinning of the glass the glass tube is drawn slightly to start the reduction and then paddled into the desired shape with a carbon paddle. As the drawing down continues the tailstock is gradually moved towards the lathe head (left) to allow glass of standard-wall thickness to be formed into the rounded portion. When closed end tubes are desired, a hand torch may be used for melting the last section of the glass, completing the separation, and fusing the tip of glass into the rounded bottom. The lathe-burner support should be adjustable to allow the burner to be tilted towards the work. The flame from a burner which may be focused this way will completely envelop the rounded end. This even heating will result in a smoother, more evenly rounded closed end. For lathes with burner-supports which do not allow focusing the flame, rounded ends may be achieved by moving the burner back and forth under the rounded section until it is evenly heated.

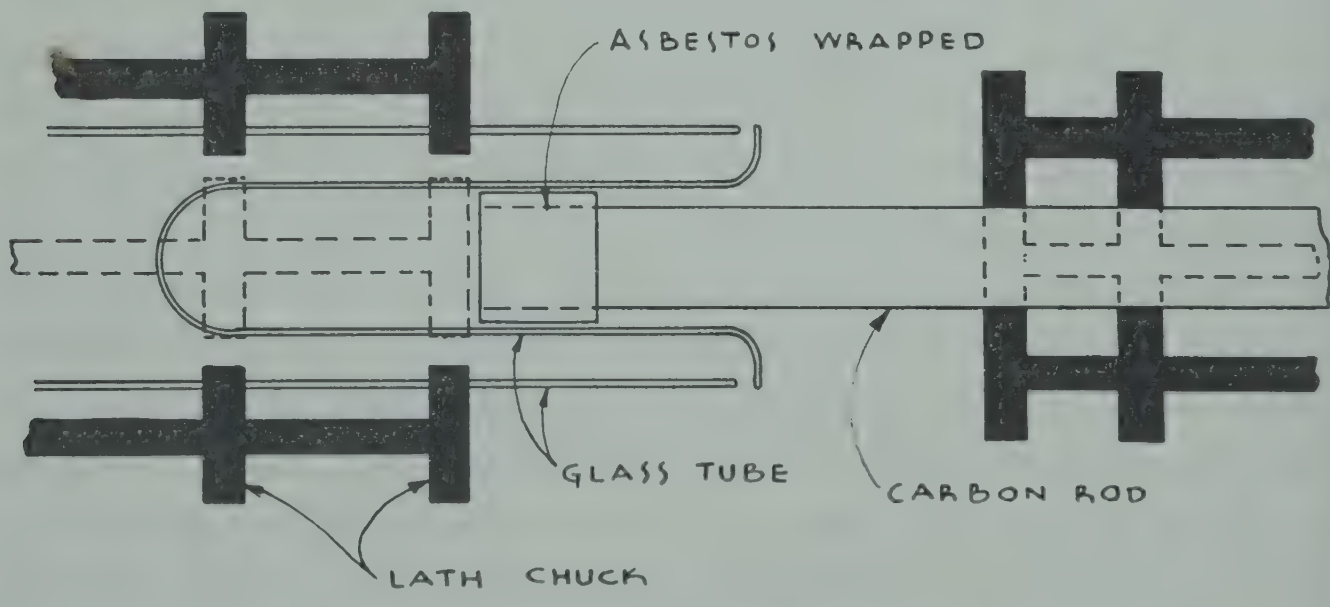

Figure 21. Making Dewars on a lathe.

Making Dewars on a Lathe. Dewars may be made quickly and easily on a glassblowing lathe if the inner tubing is prepared as described on page 84. The large piece of glass tubing which forms the jacket of the Dewar is supported in the head chuck while the inner section is supported on a carbon mandrel which is held by the chuck on the tailstock. The two pieces are assembled as shown in Figure 21 and the Dewar seal made by playing the burner flame on the edge of the seal. Commercial Dewars are made in this fashion, many times with no blowing on the inside. A smoother seal of even wall thickness may be obtained if some blowing is directed inside the Dewar to help form the seal.

Flares Made on a Lathe. Glass tubing on which the end has been cut and ground square, or tubing which has been fire-cut in the lathe, may be flared by simply increasing the speed of rotation of the chucks to the point where the glass flares by itself when it is heated. This produces a flare which is at right angles with the axis of the tube.

Flares of a specific size and angle may be made by means of a carbon rod. With the glass held and rotated in the lathe, the correct amount of glass is heated and the carbon rod used to shape this to the required depth and diameter. Should the flare become too large the diameter may be decreased by reducing the speed of rotation of the lathe and the edge of the flare heated either with the lathe burners or by means of a hand torch.

Cells with Optically Clear End Windows

Cells with optically clear windows at each end may be made by several methods. However, in each case the windows should be cut from a clear piece of Pyrex plate glass by means of the method described on page 155. To preserve the optically clear surface of the windows, it is important that the grinding be done through three pieces of plate glass waxed together as described in the same section. The tube to which the window is to be sealed should be prepared in advance by cutting or grinding both ends flat and at right angles with the axis of the tube. A side T or outlet tube should be sealed in place before either of the end seals is started.

Sealing Procedure. 1. By Hand. The window may be sealed in place by means of a sharp oxygen-gas flame from a hand torch while the tube is supported vertically with the window in position on the squared end. Care should be exercised to avoid heating the side of the tubing excessively, since, should a section of the tube become too soft, it will cause the window to move from the desired 90° position in relation to the axis of the tube (Figure 22A).

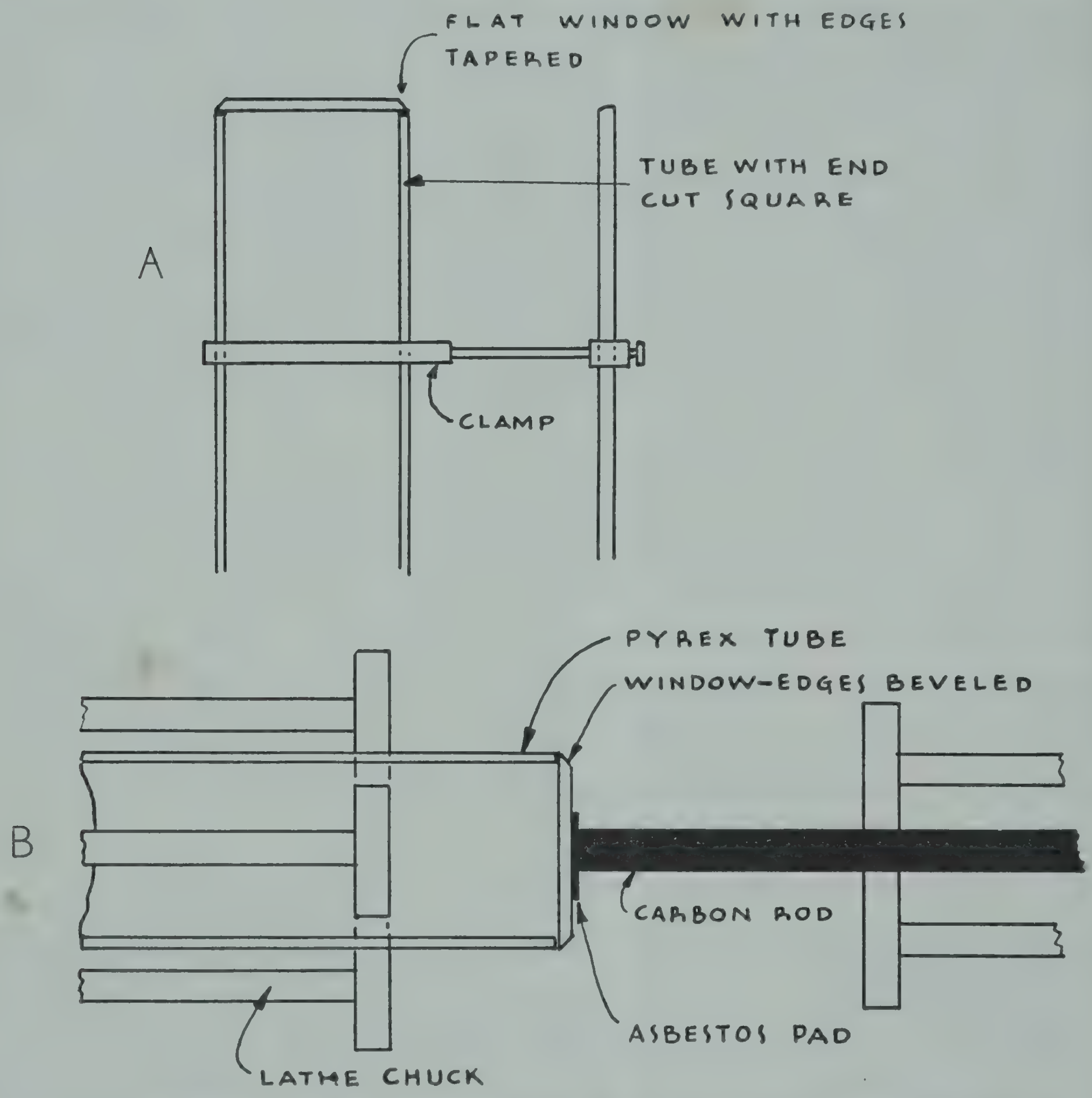
FLAT WINDOW WITH EDGES TAPERED
TUBE WITH END CUT SQUARE
A
CLAMP
PYREX TUBE
WINDOW-EDGES BEVELED
B
CARBON ROD
ASBESTOS PAD
LATHE CHUCK

Figure 22A and B. Making cells with optically clear windows.

2. Cells with Optical Windows Made on a Lathe. The tubing with the ends prepared square and the outlet arm sealed in position should be supported in a chuck of the lathe as shown in Figure 22B. The window is held in position by pressure from a carbon rod supported in the tailstock chuck. It is important that the end of the carbon rod used be perfectly smooth and cut at exactly 90° to the axis of the rod. The glass surface of the window

may be protected slightly by the use of a piece of smooth asbestos paper between the end of the carbon rod and the glass. Heating should be by means of a small hot flame from a hand torch, focused to heat only the window where it is being fused to the tube. Too large a flame allows the surface of the window to become soft and increases the danger of impressing the carbon rod or asbestos paper into the surface of the glass.

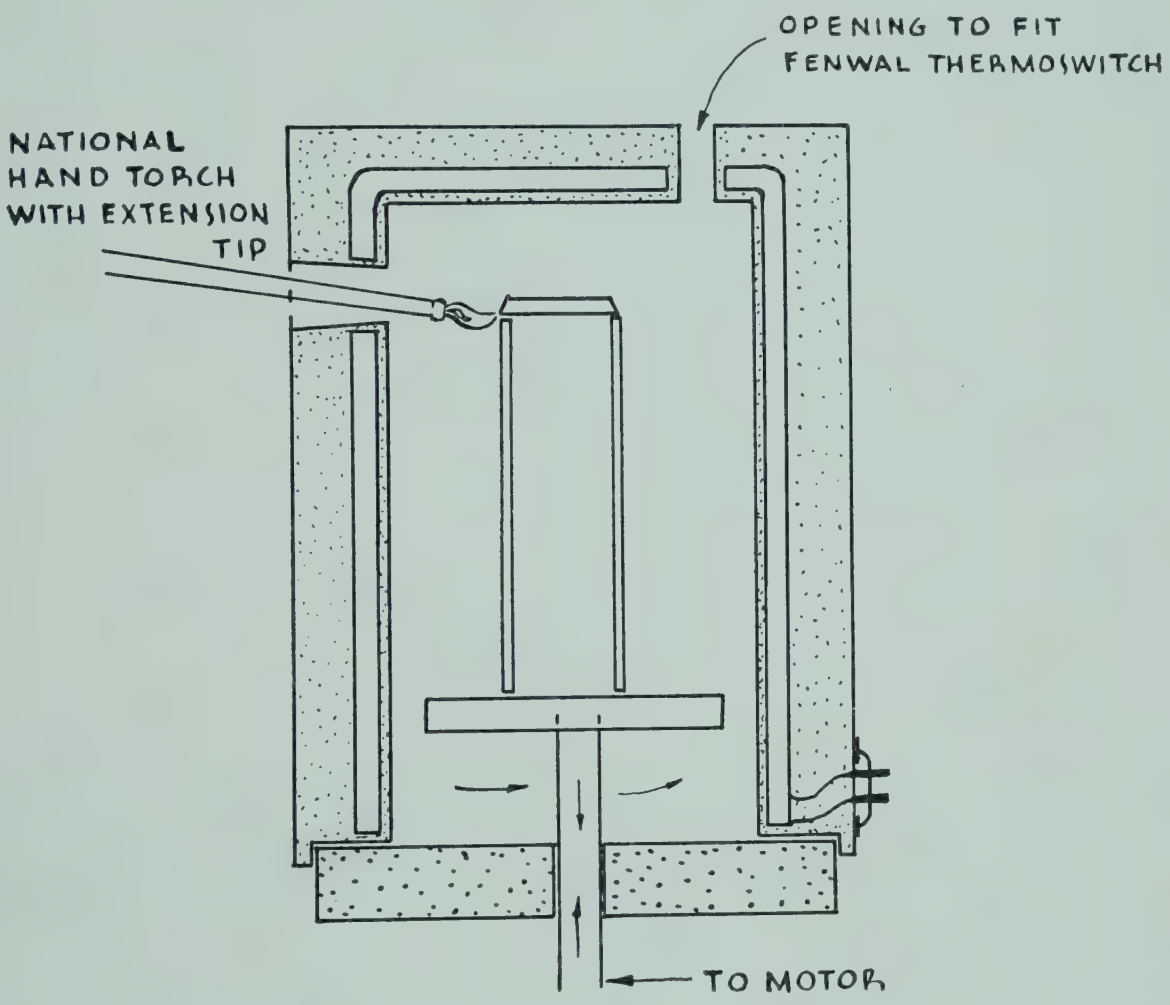

Figure 22C. Oven for making optically clear windows.

3. In an Oven. The oven shown in Figure 22C is designed specifically for making clear-windowed cells, since the floor of the oven is connected to a motor and acts as a slow turntable. In addition it may be raised or lowered to bring the top of the cell in line with the small side trap door.

A cell is made by centering a tube (prepared as previously described) on the turntable. The window is placed in position and the turntable adjusted to the correct height. The oven heat should then be turned on, and when annealing temperature is nearly reached, the window is fused to the tube by means of a flame from a hand torch directed at the glass through the trap door. After the annealing cycle is completed and the glass cooled, the cell should be turned over and the operation repeated for the window on the opposite end.

Electronic Welding of Glass

Whereas glass is a nonconductor of electricity when it is cold, heated glass is a very good conductor. This happens at a temperature below the melting point of glass, which fact lends itself to the electronic welding of glass. According to Guyer,[4] glass may be heated two ways: (1) by means of a suitable flame directed at the surface of the glass; (2) by means of internal transformation of electronic energy into heat within the glass itself. With this latter method, surface boiling and loss of volatile constituents in the glass are eliminated.

There are three methods of electronic heating.

1. By dielectric loss between the capacitor plates.
2. By eddy-current induction coils.
3. By special gas flame electrodes.

Superficially, electronic welding of glass resembles arc welding. A pin-point oxygen-hydrogen flame is often used as the electrode. Being small and extremely hot the oxygen-hydrogen flame preheats the glass at the restricted area to which it is directed. In addition the flame also serves as a gaseous conductor for the high-frequency currents. Because the flames are small and pointed they make control of the direction of the high-frequency current more accurate. The flame electrodes may also be used as a variable resistor, since by moving the tip closer to or further away from the work it is possible to exercise some control over the flow of current.

Several other advantages of electronic welding which have not been mentioned are the following:

1. The fact that adequate penetration of heat into the thick sections of glass is assured.

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[4] Guyer, E. N., *Electronics,* **18,** 92 (1945).

2. Higher melting speeds.
3. Accurate control of area heated and accurate temperature control.

One distinct disadvantage of electronic welding is the initial cost. Unless there is considerable work which may be done this way, the advantages may not offset the added cost.

VII

GRINDING GLASS

History does not record where or when glass grinding began, but evidence has been uncovered of the use of lenses of glass and rock crystal in antiquity. It is certain, though, that the art of grinding and polishing glass was highly developed in the 13th and 14th centuries. Again, there is no record to show who ground the first glass joint for laboratory use. Occasionally in the laboratory notes or biographies of early chemists a reference is made to ground joints. In around 1648, for example, von Guericke, the inventor of the air pump, says he "generally used a glass vessel with a wide neck attached by a ground joint" in his study of a vacuum.

THE MANUFACTURE OF GLASS-GROUND JOINTS IN AMERICA

The manufacture of glass-ground joints did not begin in America until the advent of World War I forced American chemists to turn to American manufacturers for both chemicals and scientific apparatus. The first glass joints of American manufacture were made in about 1918 and had a Morse taper ratio of 1 to 8, the same taper commonly used in the machine industry. Following the successful introduction of these American interchangeable joints, other manufacturers began to make similar joints. Unfortunately, no attempt was made to standardize in either size or taper, and soon it became apparent that some definite standards would have to be adopted. Under the guidance of the National Bureau of Standards, a conference was held December 19, 1929, at which time the first commercial standard, as applied to interchangeable ground joints, was established. This was adopted by mutual consent of the glass manufacturers, the dealers, and the chemists represented. Joints and stopcocks made according to these specifications are interchangeable and are marked with the symbol $, referring to a combination of S (standard) and T (taper).

SPECIFICATIONS FOR INTERCHANGEABLE $ GLASS-GROUND JOINTS

All interchangeable joints, stopcocks, and stoppers shall have a taper of 1 mm. ±0.006 mm. for each centimeter in length. To ensure this conformity, all joints and stopcocks marked $ are made and checked with standardized working tools which, in turn, are checked with gages certified by the National Bureau of Standards.

Standard taper joints come with the ground section in any one of three lengths, the standard or full length, which is the most widely used, medium length, and short length. Standard taper joints are designated by figures which indicate (in millimeters) the diameter of the large end and the length of the grind. Thus $ 5/20 indicates a ground section 5 mm. in diameter at the large end and 20 mm. long. See Table I.

USE OF $ GLASS-GROUND JOINTS IN MODERN RESEARCH

The use of ground joints and stopcocks on glass apparatus has become standard procedure; in fact without these ground joints and stopcocks, chemists would have a difficult time working with gases and liquids. Much of the standard laboratory equipment has been fitted with ground joints to facilitate cleaning, and in many cases it is so designed that by changing auxiliary parts, apparatus for different experiments can be assembled.

Until a few years ago it was thought by some research workers that commercial ground joints and stopcocks were not satisfactory. This was especially true in regard to high-vacuum work; however, the great advances in the art of grinding these inter-

TABLE I. Specifications and Designations for ₮ Ground Joints.

When the National Bureau of Standards, through its Committee on Interchangeable Ground Joints introduced a new series of Medium Length Joints, it made necessary a new method of designation, consisting of two figures, the first indicating the approximate diameter (in millimeters) at the large end, the second giving the length of ground glass zone. Thus ₮ 5/20 indicates a ground zone 5 mm in diameter at the large end, and 20 mm in length. For purposes of comparison, old and new designations for full length joints, the designations used for medium length and for the newly introduced short length (see Commercial Standards CS21-39) joints are shown below:

(Taper, 1 to 10; i.e., one mm ± 0.006 mm per centimeter of length on diameter)

FULL LENGTH JOINTS			MEDIUM LENGTH JOINTS			SHORT LENGTH JOINTS		
†Old Designation (Approx. Diam. Small End) mm	New Designation (Large Diam./Length of Ground Zone) mm	Computed Diam. Large End of Ground Zone mm	Designation (Large Diam./Length of Ground Zone) mm	Computed Diam. Large End of Ground Zone) mm	Approx. Diam. Small End of Ground Zone mm	Designation (Large Diam./Length of Ground Zone) mm	Computed Diam. Large End of Ground Zone mm	Approx. Diam. Small End of Ground Zone mm
3	5/20	5.0	5/12	5.0	3.8			
5	7/25	7.5	7/15	7.5	6.0			
7	10/30	10.0	10/18	10.0	8.2	△10/10	10.0	9.0
9	12/30	12.0	12/18	12.0	10.2	△12/10	12.0	11.0
11	14/35	14.5	14/20	14.5	12.5	△14/10	14.5	13.5
15	19/38	18.8	19/22	18.8	16.6	△19/10	18.8	17.8
20	24/40	24.0	24/25	24.0	21.5	△24/12	24.0	22.8
25	29/42	29.2	29/26	29.2	26.6	△29/12	29.2	28.0
30	34/45	34.5	34/28	34.5	31.7	△34/12	34.5	33.3
35	40/50	40.0	40/35	40.0	36.5	△40/12	40.0	38.8
40	45/50	45.0				△45/12	45.0	43.8
△45	50/50	50.0				△50/12	50.0	48.8
50	55/50	55.0				△55/12	55.0	53.8
55	60/50	60.0				△60/12	60.0	58.8
65	71/60	71.0				△71/15	71.0	69.5

△Newly introduced ground joints. These sizes were not standardized when old designations were used.

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†The old designation also served to denote the approximate outside diameter of the straight section of tubing forming a

changeable surfaces have produced commercial joints of high quality, as well as precision-ground high-vacuum stopcocks that give excellent results. Sheppard and Lebig[1] have found that with a precision-ground stopcock which has been properly lubricated with a suitable high-vacuum stopcock grease, no difficulty due to stopcock leaks will be experienced. They state that in their laboratory several systems containing stopcocks maintained a vacuum, as indicated by a barometric manometer (accurate to 0.2 mm.), for two years without any pressure change. In another experiment an apparatus in constant use, containing paraffin hydrocarbon gases whose vapor pressures were 0.0001 mm. or less at 210° C. and involving 17 plain stopcocks without mercury seals, consistently failed to show pressure increases greater than 0.0001 to 0.0005 mm. over a period of two to four weeks, as measured by a McLeod gage.

With the high quality of commercial $ ground joints and stopcocks available, and the variety of precision-ground stopcocks which may be purchased, it is seldom necessary for a research glassblower to fabricate his own glass joints and stopcocks. In view of this, a discussion of the actual gathering of the glass and tooling or shaping blanks for interchangeable joints and stopcocks will be omitted. For those particularly interested in this phase of glassblowing, a detailed description is given by Barr and Anhorn.[2]

GRINDING TOOLS MADE FROM A JOINT

Because of the extensive use of ground-glass equipment, a knowledge of repairing and regrinding damaged ground surfaces is essential. For grinding involving only one joint, it is possible to use a matching new joint as the grinding tool. When another $ joint is to be used for the grinding tool, it is important that the joint be new or in excellent condition and, unless the two joints

[1] Sheppard, M., and Lebig, P. G., *Ind. Eng. Chem.*, **19**, 1059 (1927).

[2] Barr, W. E., and Anhorn, V. J., *Scientific and Industrial Glassblowing and Laboratory Techniques*, Instruments Publishing Co., Pittsburgh, 1949.

ground together are to be used as a pair, it is advisable to discard the unused joint, since it will not be interchangeable with other \$ joints. This practice of regrinding joints, using other \$ glass joints as grinding tools, is practical only when damage to the joint being repaired is slight and a light touching up of the ground surface is all that is necessary.

SETS OF METAL GRINDING TOOLS

If several joints are to be repaired or if over a period of time several sizes of joints may be reground, it will be advantageous to have two sets of grinding tools made up for each size of ground joint for which the need of grinding is expected. For high-precision grinding, as with vacuum stopcocks and joints, a third set of grinding tools is recommended. A set of tools consists of a taper grinder and a taper socket grinder of each joint size.

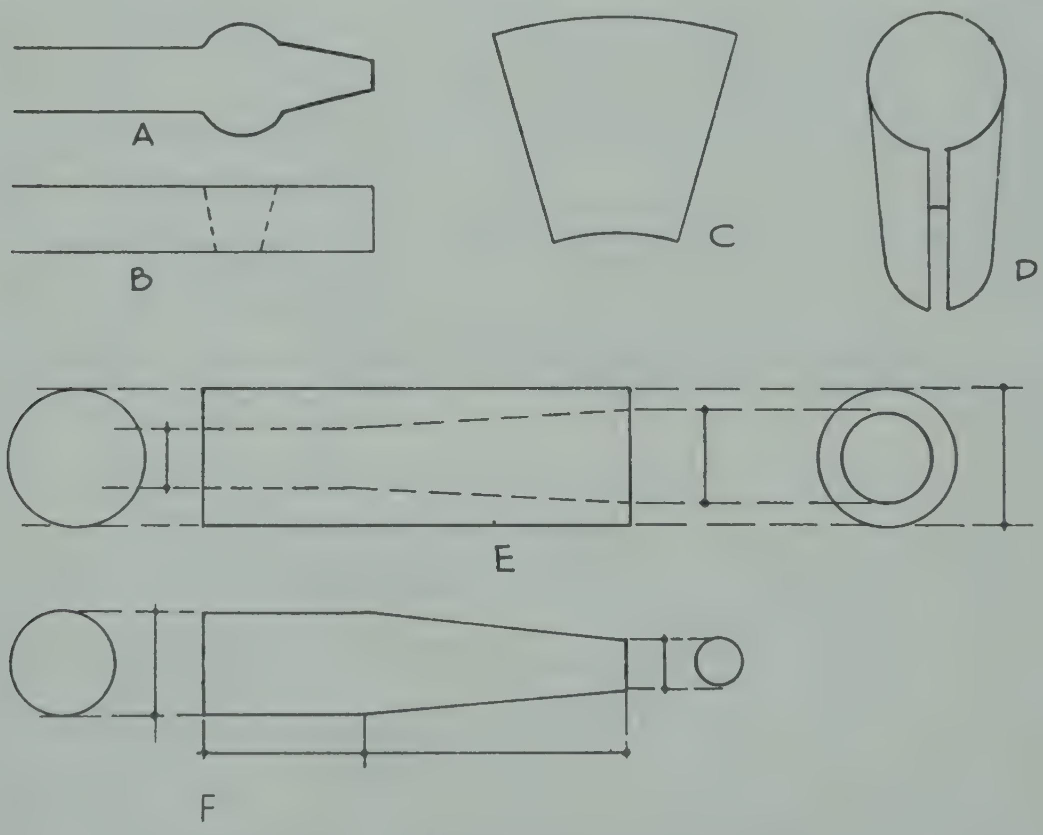

Figure 1. Metal grinding tools.

Grinding tools may be made of either brass or steel. Tools made of brass are good for grinding about 20 joints while those of steel may be used for about 40 joints. Steel grinding tools which have been casehardened may be used for an infinite number of finished joints. The useful period of each tool may be prolonged by using it for the prescribed number of joints with the finest mesh Carborundum and then for the same number of joints with the next courser grit and so on to the rough grind. When 600 mesh Carborundum is to be used for making precision high-vacuum joints, four sets of grinding tools should be available. However, the first rough grind may be started with the grinding sleeves shown in Figure 1. These sleeves are made from a piece of fairly soft metal and are bent to shape around a taper grinder as shown in Figure 1A and 1B. A rough grinding sleeve for the outer joints may be formed around the taper grinder, as shown in Figure 1C and 1D.

Figure 1E and F shows a set of grinding tools for the finished grinds in both inner and outer joints. Exact dimensions are not given, since these measurements may be obtained from $ glass joints. The length of the grinding surface for the taper grinder socket should be 15 mm. longer at the small end than the length of the glass joint, to allow for the resurfacing of the tool at intervals. The grinding taper should also be longer, but at the large end, to allow future resurfacing.

When grinding is done intermittently, a record may be kept on each tool by marking in pencil the number of joints ground with each tool as well as the mesh of the Carborundum used.

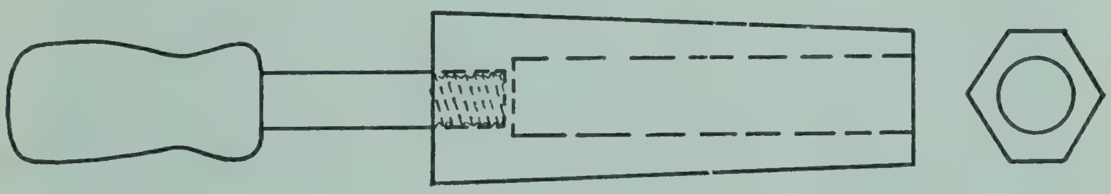

Figure 2. Steel reamer or shaper.

In extreme cases of joint distortion, a standard taper hexagonal reamer may be necessary to reshape the joint before actual regrinding begins. Carbon hexagonal shapers with the $ 1 : 10 taper may be purchased from glassblowing supply houses. Figure 2 shows a similar $ shaper made from steel. The advantages of a steel shaper are obvious in that it is unbreakable and will not wear out. Before use these steel reamers should be given a coating of Aquadag.

SELECTION OF ABRASIVES FOR GRINDING

The choice of abrasives in grinding interchangeable joints determines the fineness and perfection of the ground surface.

Rough grind	Carborundum No. 80
Second rough grind	Carborundum No. 150–180
Commercial finished grind	Carborundum No. 400
High-Vacuum finish grind	Carborundum No. 600

GRINDING EQUIPMENT

The actual grinding can be done easily if the grinding equipment shown in Figure 3 is available. If not, a vertical drill press may be used. More care must be observed in grinding glass joints in a drill press, since the vertical position allows the Carborundum and water to run out of the joint, leaving it dry. A machine lathe may be used, but this is not recommended unless a good covering can be devised to protect the ways and gears from getting Carborundum on them. Regrinding may be done by hand, although this is a very slow and laborious process, especially if several joints are to be reground.

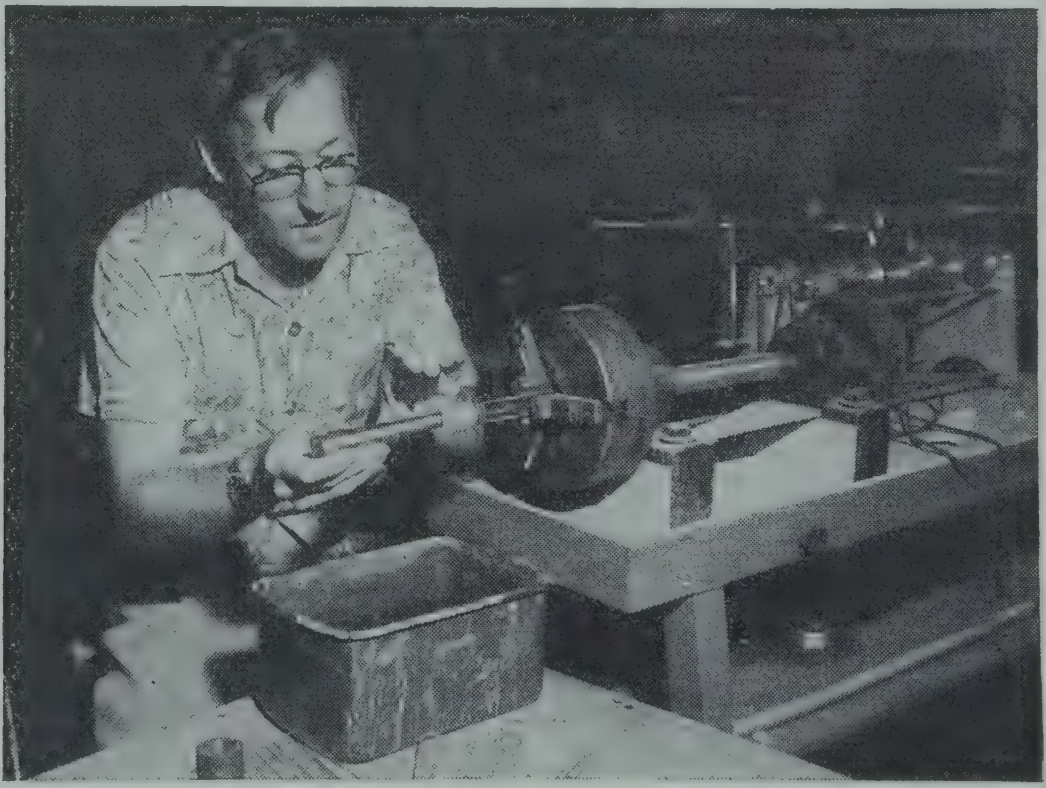

Figure 3. Grinding head.

GRINDING PROCEDURE

During grinding, a diminishing cutting sound indicates the need for additional Carborundum. The ground surfaces should be inspected each time before adding new Carborundum to prevent unnecessary grinding away of the joint. A pencil mark the full length of the ground surface will enable the grinder to follow the progress of the grinding. When the pencil mark has completely disappeared, the joint should be given a few seconds additional grinding and then thoroughly washed preparatory to going on to the next finer grade of Carborundum.

TESTING REGROUND SURFACES FOR LEAKAGE

A simple test for the accuracy of the grinding is made by thoroughly cleaning both ground surfaces, drying the outer member, and then inserting the inner member while it is still wet. If the inner member is pushed to one side, any movement between the two surfaces will show up in movement of the water contact. An abnormal movement of this water contact indicates either that the grinding is too hurried or that the grinding tools need resurfacing.

SPHERICAL BALL AND SOCKET JOINTS

More recent developments in the interchangeable glass ground joints are the spherical ball and socket joints which are now on the market and which are enjoying increasing popularity and usage. Ball and socket joints are designated by two numbers; the first indicates the diameter of the ball in millimeters, and the second indicates the inside diameter of the tubing in millimeters. Thus, a 28/12 has a ball 28 mm. in diameter and is made from 12 mm. I.D. tubing. Regrinding procedure for spherical joints follows, in general, that given for $ joints. The one notable exception is that the nonrotating member should be moved from side to side during grinding so that grinding is even over the whole surface of the ball and socket.

GLASS CHECK VALVES

Glass check valves are used for stopping the flow of liquids and gases. The precision of the grind necessary will depend on the use for which the valve is intended. As is true with ground joints, the best results are obtained when a special tool is used for the actual grinding rather than grinding the check ball into the seat. Satisfactory check valves for many operations may, however, be made by grinding the check directly into the valve seat. In Figure 4 are shown several different types of check valves. Figure 4A shows a mercury-filled valve that drops down to shut off. These valves are used in automatic Toepler pumps and are closed when the mercury level drops. The check valve shown in Figure 4B is also used in a Toepler pump but it closes when the level of the mercury rises. The valves shown in Figure 4A and B are made by grinding the valve seat with a steel ball bearing sealed on a handle, as shown in Figure 4C, and using three grades of Carborundum as in grinding $ joints. The ball bearing may be used more than once by recementing it to the handle with a new surface exposed. The float is then made and ground into the prepared seat, using only the final two grades of Carborundum. The extension or open end tubes form a handle in grinding these checks into the valve seat. Float A is then filled with mercury and sealed off, while Float B is sealed off empty since it operates as an actual float. Both check valves are then sealed into position by making the second reduced seal, after which three indentations are pushed in to keep them centered in position (see Figure 4E). Figure 4D and E shows two other types of check valves. The check shown in Figure 4D shows a solid glass ball and is especially good for use when standard-wall tubing is sealed to capillary tubing. For grinding the seat for this valve, a special tool shown in Figure 4G is used. The grinder should be made either of brass or steel and shaped to an accurate 45° angle. Several of these tapered grinders should be made so that the taper used for the finest grind is used only once or twice. It may, however, be used again on each of the coarser grinds before being resurfaced. Figure 4F shows the valve seat after grinding.

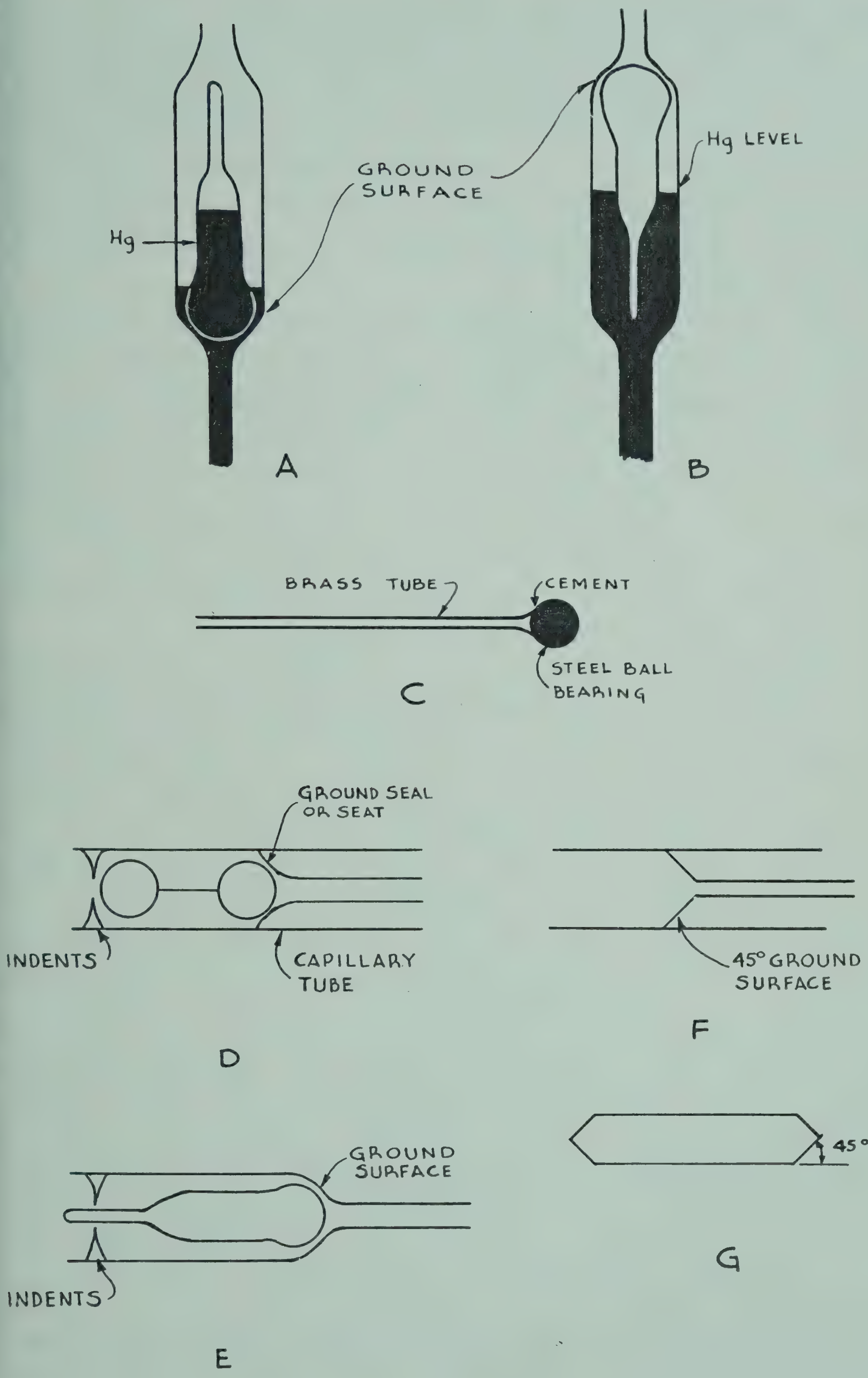
GROUND SURFACE
Hg
Hg LEVEL
A
B
BRASS TUBE
CEMENT
STEEL BALL BEARING
C
GROUND SEAL OR SEAT
INDENTS
CAPILLARY TUBE
D
45° GROUND SURFACE
F
45°
GROUND SURFACE
INDENTS
E
G

Figure 4. Glass check valves.

Glass Ball Check Valves

Glass ball bearings are available commercially from the Hartford Steel Ball Company,[3] which manufactures balls either of Pyrex or soft glass in many sizes, starting at $^1/_8$ in. up to 1 in. O.D. These glass balls have a spherical tolerance of 0.0002 in. and a size tolerance of —0.001 in. However, when only a few glass ball checks are desired, they may be made and ground by the following procedure.

If a glass rod is rotated with the tip of the rod in a flame, a ball of glass will be gathered at the end of the rod. This rotating and heating should be continued until a ball of the desired size has been gathered. The glass will form into a ball more quickly and the ball will be more symmetrical if the rotating is done with the rod held in a nearly vertical position. When the desired diameter has been obtained, the axis of the ball may be brought in line with the axis of the rod by rotating in a horizontal plane and continuing the rotation until the glass has set.

The ball is finished by heating a small rod of glass and sticking it lightly to the ball, after which the original rod or handle is cut away. With the newly attached rod as a handle, the unfinished side of the ball is heated and worked into a smooth shape by rotation. Should the ball appear too short and thick, it may be made more spherical by being rotated in a vertical position with the ball hanging down. On the other hand, if the ball is too elongated, it may be brought back into shape by being rotated vertically with the ball up. When the glass is cool, the temporary handle is broken from the ball, and if it was not too hot when it was attached, it will break away without causing serious damage to the surface of the ball. The surfaces of the ball are ground by placing several balls between two pieces of plate glass which are pushed together and rotated with a circular motion. The bottom plate should be covered with wetted Carborundum.

Glass Ball Check Valves on Stems

Figure 5 shows a type of glass check valve commonly used in controlling distillate take-off in many types of electronically controlled still heads. These are made by the same gathering procedure as described in the preceding paragraph. The glass ball should be gathered to the same size as the ballbearing which will be used for forming the grinding template and also for grinding the valve seal.

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[3] Hartford, Connecticut.

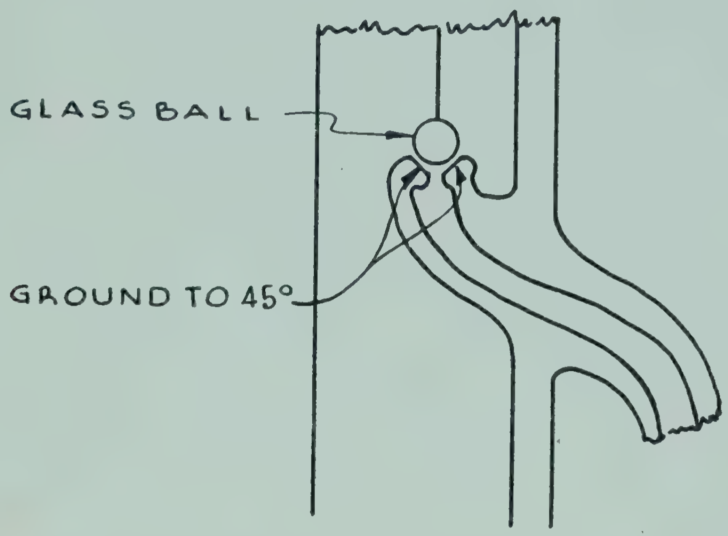

Figure 5. Glass check valves on stems.

Two pieces of soft brass or copper (copper preferred) are used to form the grinding mold. These should be at least ½ in. thick or thicker, depending on the size of the ball bearing to be used. They are heat-treated to soften them and then both pieces are placed in a vise with the ball bearing between them, as shown in Figure 6A. When the ball is imbedded as shown in Figure 6B,

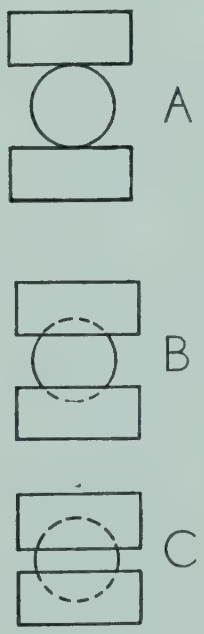

Figure 6. Preparing mold for grinding ball checks.

the first steel ball is removed and a new ball is inserted before continuing the clamp the brass or upper pieces together as shown in Figure 6C. The second ball will insure the mold's being more nearly perfect, although if the blank has been sufficiently softened, the mold may be finished with the original steel ball. Care should be observed in the final clamping not to imbed the ball past the center mark in either mold. Otherwise removing it without damaging the mold will be difficult, if not impossible.

One of these grinding molds should be made for each grade of Carborundum to be used, and they may be re-used by starting at the finest grade of Carborundum and working down to the coarser, as described for other grinding tools. As a precaution, the grinding mold should be discarded when it begins to show signs of excessive wear, even though it may not have been used for all the different grits.

Satisfactory check valves may be obtained with only three grinding steps, beginning with 100 mesh and 150 to 180 mesh for the intermediate grind and using 400-mesh Carborundum for the last grind. However, for high-precision ground check valves, 600-mesh Carborundum and a final polish with optical grit #302 are recommended.

When highly viscous materials are to be run through a check valve, sticking may occur when the valve seats are ground with ball bearings. This sticking can be virtually eliminated if the valve seat is ground with a special 45° angle tool as shown in Figure 4G. Small microspherical joints which may be used as check valves can be obtained from Ace Glass Company.[4]

REGRINDING STOPCOCKS

Commercial-grade stopcocks are not intended for high-vacuum work and should be used in vacuum systems only as a last resort. When they are used, they should be given a fine grind using optical Carborundum #302, grinding the plug into the barrel. A stopcock so ground, unfortunately, loses its interchangeability with other stopcocks marked $.

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[4] Vineland, New Jersey. (Catalog No. 7661.)

When the barrel of a stopcock marked $ is broken, it is good practice to save the plug, since broken $ plugs in other stopcocks may be replaced by any plug in which the holes line up. When it becomes necessary to grind a different stopcock barrel and plug together, a plug should be chosen that gives a 1 to 2 mm. grind-away before the holes in the shell and the plug line up. The initial grind is begun using 220-mesh Carborundum, and 320-mesh Carborundum is suitable for the intermediate grind. When the holes in the barrel and plug practically match, grinding is finished with 400-mesh Carborundum. Stopcocks to be used for vacuum work will need a final grind with 600-mesh Carborundum.

Actual hand grinding is performed by turning the inner member a few complete turns and removing it to reseat it, before making a few more complete revolutions. On occasion, after removing and reseating the inner member, it is good practice to make a few revolutions backwards from the preceding revolutions. The repeated removal of the inner member and reseating it prevents the Carborundum from becoming streaked and grinding grooves in the stopcock surface. It also allows the operator to observe the dampness or dryness of the grinding compound. It is very important that the Carborundum and the ground surfaces be kept wet during the grinding. If the Carborundum becomes dry during the grinding and "freezes" the plug in the barrel, it is almost impossible to remove the plug.

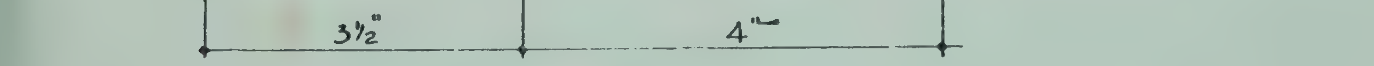

Figure 7. Stopcock plug holder.

If a number of stopcock plugs are to be ground, the holder shown in Figure 7 should be made, and it will be necessary to make a separate holder for each size of stopcock to be ground.

Final forming of the holder to fit each size of plug is obtained by heating the handle of a discarded plug, clamping it in position, and allowing it to burn the wood until a good fit is obtained.

DRILLING SMALL HOLES IN GLASS

In Pyrex glass apparatus which can be heated, small holes may be drilled by using a piece of heated tungsten wire held in a pin vise (see Figure 8) and rotated by hand, since high speed is not necessary for the successful drilling operation. Actually the drilling motion need not be continuous in one direction, but may be a few revolutions one way and a few in reverse. If many holes are to be drilled, however, a small chuck on an electric motor will be desirable for holding and turning the tungsten wire.

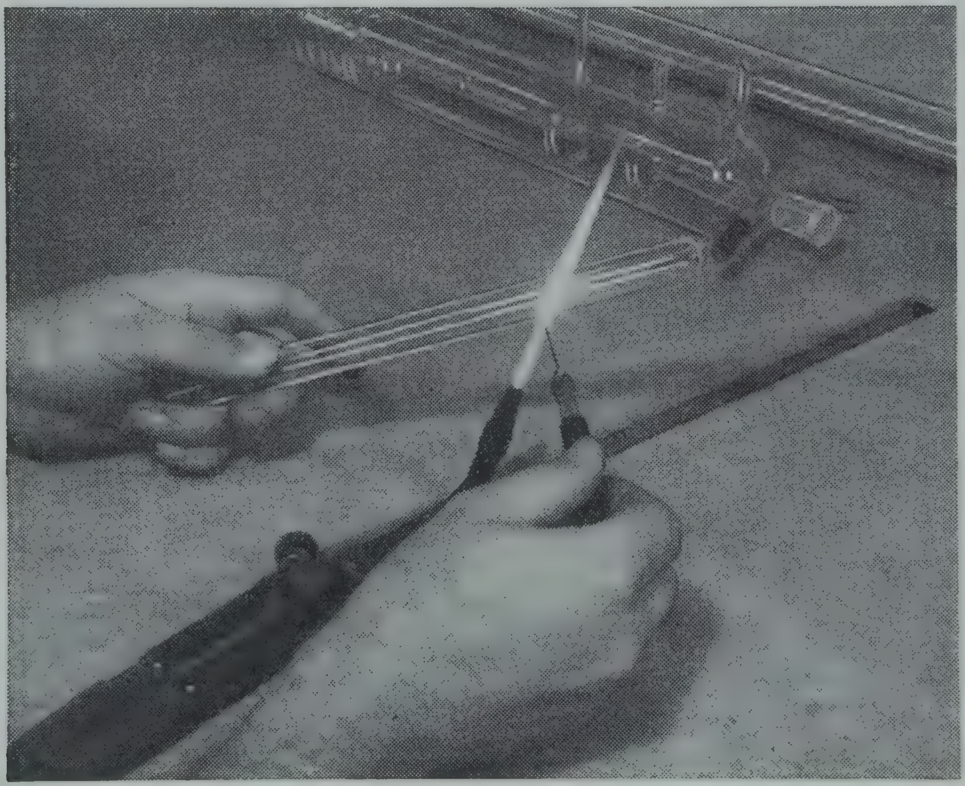

Figure 8. Drilling holes in glass with a hot tungsten wire.

GRINDING HOLES IN GLASS

Grinding Small Holes in Glass

Glass which can not be heated can have holes drilled in it by using the special steel drill shown in Figure 9C. The drill can be made from a piece of hardened steel, drill rod, or a triangular file.

After being formed the drill should be hardened by being heated and quenched in mercury. The drill is ground so that one point is longer than the other two, this point forming the cutting edge and moving at the circumference of the hole being drilled. For the best results the glass should be drilled first by countersinking on both sides, with the tool shown in Figure 9A, and then drilled from both sides so that the hole is not completed on either surface.

During the drilling sufficient pressure should be applied to the drill to keep the tool cutting; however, this pressure must not be so great as to crush the glass. A solution of turpentine and camphor, containing enough camphor to make it fairly viscous, makes a suitable lubricant for grinding. A 50-50 mixture of soybean oil and kerosene also makes a satisfactory lubricant for the grinding.

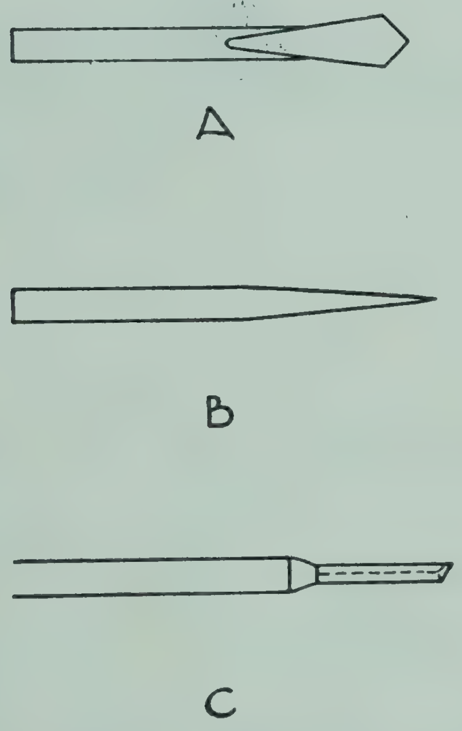

Figure 9. Tools for grinding small holes in glass.

The tool shown in Figure 9B is a reaming tool for smoothing or enlarging the diameter of the hole after it has been drilled.

Grinding Large Holes in Glass

Large holes in glass may be ground by means of the special cutters shown in Figure 10. This may be made up to any desired size, of either brass or steel. A mixture of coarse Carborundum

(100-mesh) and water gives reasonably fast grinding, and this is held in place during the grinding by means of a small clay dyke formed around the intended hole.

When holes are being ground in pieces of flat glass, a smooth cut without jagged edges on the bottom side may be obtained by cementing three pieces of glass together using beeswax, as shown in Figure 10A, and drilling through the first two and part way into the third. The top piece of glass protects the surfaces of the piece to be used from being scratched by the swirling Carborundum during the grinding, and the bottom piece prevents the grinding tool from breaking out through the glass and leaving jagged edges.

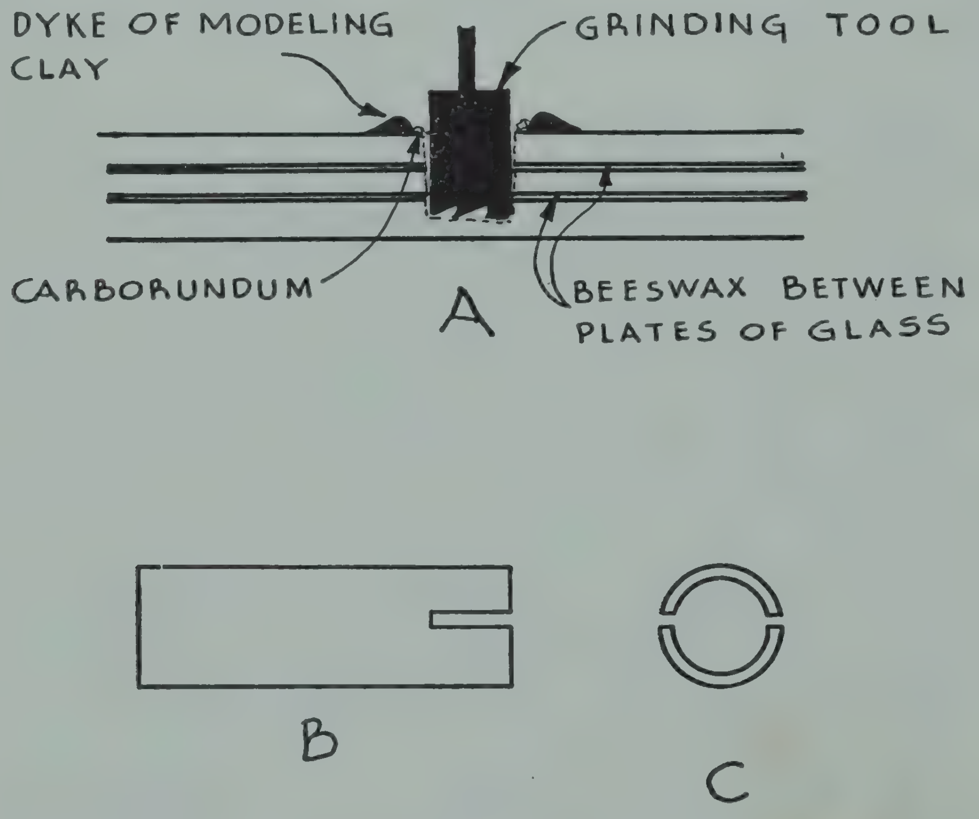
DYKE OF MODELING CLAY
GRINDING TOOL
CARBORUNDUM
A
BEESWAX BETWEEN PLATES OF GLASS
B
C

Figure 10. Tool for grinding large holes in glass.

VIII

GLASS TO METAL SEALS

Glass to metal seals are an important step in present-day scientific glassblowing. Under the proper conditions these seals are as easy to make as glass to glass seals; however, before any glass to metal seals can be attempted the physical characteristics of both the glass and the metal must be known. To assure a satisfactory seal, the glass must wet the surface of the metal, and the metal must have a coefficient of expansion similar to that of the glass so that the stresses set up in the glass by the expansion and contraction of the metal will not exceed the tensile strength of the glass.

What is called glass wetting the metal is actually the glass dissolving in the oxide layer which covers the metals. Hull and Berger[1] have shown that when a glass bead is placed on a metal plate that is free from oxide, and both are placed in a furnace in a hydrogen atmosphere, no seal is obtained. Similar results were observed with an inert gas such as nitrogen or carbon dioxide. Nevertheless, when the metal was oxidized before being placed in the furnace in an inert atmosphere, a strong bond was formed between the glass and metal, thus showing the importance of oxidizing the metal before a seal with glass is attempted.

The degree of oxidation is very important. Too thin an oxide layer prevents the glass from forming a strong bond with the metal and results in a weak seal. If the oxide coating is too heavy, the glass bonds firmly to the oxide, but the danger of the heavy oxide layer's breaking away from the metal is increased and seals defective for vacuum work and mechanically weak may result.

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[1] Hull, A. W., and Berger, E. E., *Gen. Elec. Rev.*, **37,** 96, (1934).

PROCEDURE FOR SEALING GLASS TO METAL

Tungsten to Pyrex 7740

The linear coefficient of expansion of tungsten is somewhat higher than that of Pyrex Brand No. 7740 or that of Nonex 7720. However, either glass adheres to the metal. Although it is not a standard commercial procedure, tungsten can be sealed directly into Pyrex glass. This seal is suitable for vacuum, provided the diameter of the wire is 50 mil. or less and the service requirements are not too severe. To prepare tungsten for sealing, it is degassed by being heated to a dull red, then cleaned by being rubbed with a stick of potassium nitrite (KNO_2), thus dissolving the tungsten oxide. The nitrite is washed off, exposing the shining, clean surface of the wire. The tungsten is then dried and reoxidized by passing it through a gas-oxygen flame. This reoxidizing is one of the most important steps in the entire process and can be mastered only by practice. A close-fitting sleeve of glass, 10 to 20 mm. in length with a wall thickness of ½ to 1 mm., is slipped over the wire (Figure 1A). This is heated progressively from one end to the other so that the glass shrinks onto the metal, driving the gas out through the open end and thus avoiding trapping air between the glass and the metal. Since the metal in contact with the glass appears enlarged, the progress of the seal may be easily followed.

When a good seal has been made, it will have a golden yellow or reddish-copper color. Because of its higher melting point Pyrex glass requires more heating to form a strong bond with the tungsten than does some of the softer glass. If, after one pass through the flame, the seal is black, heating should be started at one end and worked down the length of the seal again. Possibly a third heating will be required to bring out the copper color, depending on the length of time used in each heating. Thus, if after three separate heatings, the seal still remains black, it is an indication that the tungsten either (1) is still dirty or (2) has been oxidized too much before starting the seal. It is not advisable to use a blackened seal.

A silver-colored seal indicates that the oxidizing before sealing was not sufficient; the glass should be removed and the complete operation repeated.)

(*Single Tungsten Wire Seals.* When the tungsten wire can be supported from the inside (i.e., when a short section of glass tubing with the tungsten seal is to be fused to a piece of apparatus

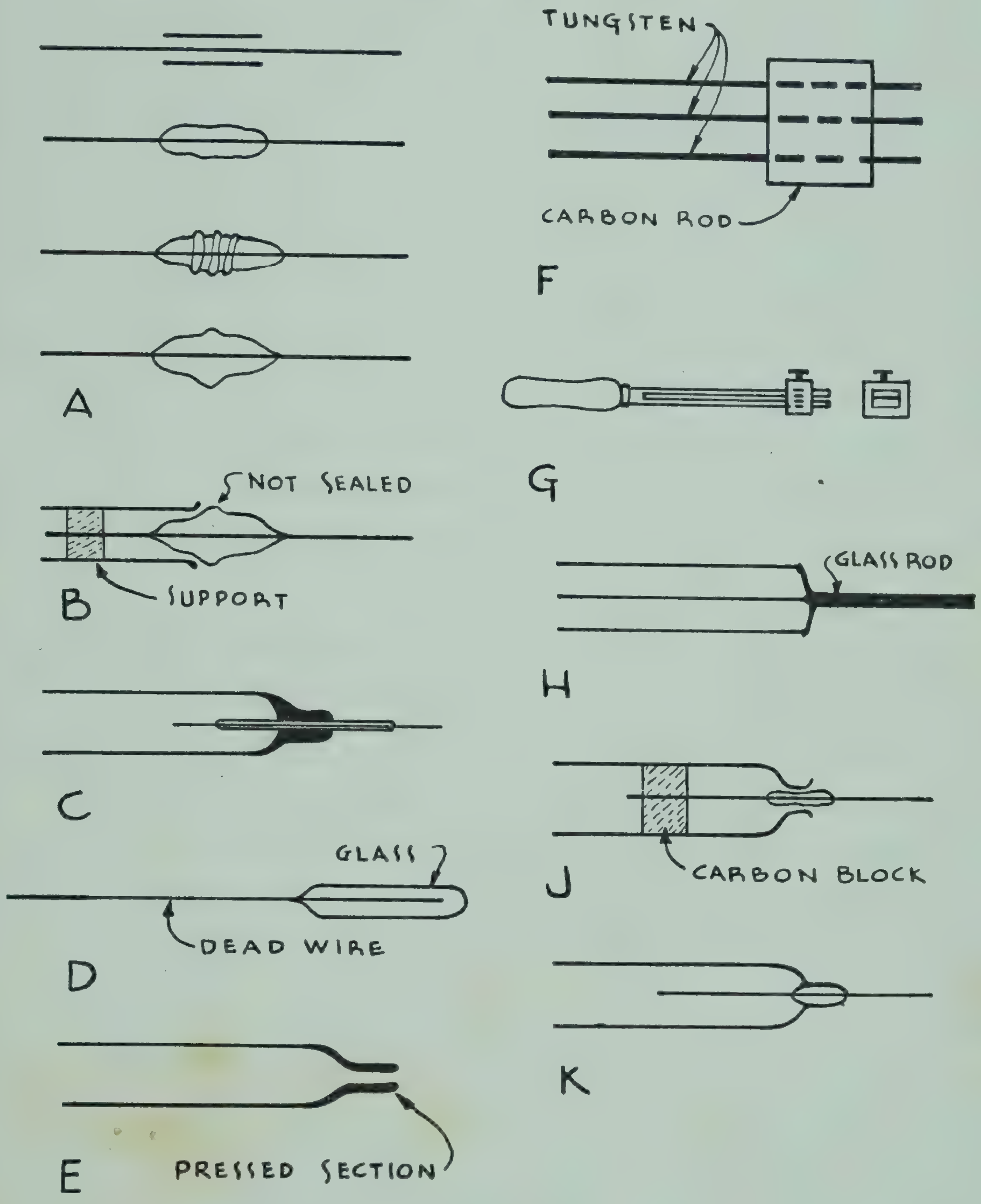
TUNGSTEN
CARBON ROD
F
A
G
NOT SEALED
SUPPORT
B
GLASS ROD
H
C
GLASS
DEAD WIRE
D
CARBON BLOCK
J
K
E
PRESSED SECTION

Figure 1. Steps for Pyrex to tungsten seal.

after the tungsten seal is completed), a piece of asbestos tape may be wrapped around the tungsten and placed in the position shown in Figure 1B. (The asbestos should be preburned to prevent contamination of the glass during the operation of making the tungsten-glass seal.) In this case no additional support is necessary, and the seal is completed by rotating the glass in a flame and allowing the outer piece to fuse to the beaded wire.

If an inside support is not feasible, the tungsten wire may be supported from the outside by means of a pin vise. The procedure in this case is similar to that used in making a simple insertion seal. The completed seal is shown in Figure 1C.

Multiple Tungsten Wire Seals. When more than one tungsten wire is to be sealed through the glass, the pinch-press method is used. In this type of seal the tungsten wire needs only a thin glass sleeve and any dead end or support wire should have the end covered with glass as shown in Figure 1D. The tungsten wires are prepared as previously described and the outer tube is prepared by closing off one end, reheating this end, and pinching it into the shape shown in Figure 1E by means of a pair of flat paddle forceps. This flattened section need not be longer than ½ in. The end of the flattened section is blown out and the inside opening carefully shaped with a carbon rod to assure room for the even spacing of all the tungsten wires.

In some types of multiple seals it is possible to support the wires from inside the tube. When this can be done, a carbon jig may be made with the required number of holes accurately spaced and drilled to hold the tungsten wire as shown in Figure 1F. The holes should be of the proper inside diameter to support the wires steadily and yet afford easy removal of the carbon jig when the press seal is completed. Multiple seals in which the tungsten wires may not be supported from the inside may be made using the jig shown in Figure 1G, or a temporary support may be fashioned from Pyrex glass rod as shown in Figure 1H. The tungsten wires should be supported so that a short section of the beaded glass extends beyond both ends of the press. The flattened section is then heated and, when it is sufficiently soft, forceps are used to press the glass against the beaded wires to form a good seal (Figure 1J). A side view of the finished multiple seal is shown in Figure 1K.

Cleaning Tungsten Wire Electrically. A saturated solution of potassium nitrite may be used for a bath for cleaning tungsten electrically. About 10 volts a.c. should be applied to the tungsten and another electrode, which may be tungsten or silver. After being cleaned and washed, the tungsten is ready to be reoxidized.

Cleaning Used Tungsten Leads. When only a few leads are involved, they may be cleaned individually by using a potassium nitrate stick and heating the tungsten in a flame. When many leads are involved, the following procedure can be used:

1. For leads without nickel sleeves, procedures (*a*) and (*b*) should be used.
2. For leads with nickel sleeves, procedure (*b*) should be used, followed by (*c*).
   - (*a*). The leads are placed in a beaker and covered with 2 to 3 in. of nitric acid. They are then heated until evolution of bubbles indicate that the acid is reacting. The heat should be adjusted and this boiling maintained for 45 minutes, after which the solution is cooled for 15 minutes. Water is then added to hasten the cooling. When cool the acid is drained off and the leads washed thoroughly with water.
   - (*b*). Enough sodium hydroxide (20 %) is put in a beaker to cover the leads by 3 to 4 in. The leads are then boiled in this solution for 1 hour and rinsed well with water.
   - (*c*). Nitric acid is poured over the leads in a beaker (at room temperature) and allowed to stand for about 1 minute, after which the acid is drained from the beaker and the leads thoroughly rinsed with tap water.

Tungsten to Pyrex 7720 (Nonex)

Tungsten larger than 50 thousandths of an inch should be sealed into Pyrex brand 7720 glass. Some glassblowers prefer to make all tungsten seals with this glass. The procedure is the same as that used for Pyrex No. 7740. The tungsten is beaded with Nonex glass and sealed into a tube of Nonex glass. Since this is a lead glass it requires working in an oxidizing flame. When the completed seal is to be attached to Pyrex No. 7740, a step seal of uranium glass No. 3321 is advisable. The recommended sealing order is tungsten, Pyrex Nos. 7720, 3321, and 7740. Nonex glass is widely used commercially for tungsten seals.

Tungsten to Pyrex No. 7991

This glass can be used for tungsten seals in place of No. 7720. It is a nonlead glass and can be worked in a regular flame. It also has a higher softening point and can be baked out at a higher temperature.

Tungsten to Pyrex No. 3321

This glass can be used for making tungsten seals and it may be sealed directly to No. 7740 Pyrex glass. The metal seal is made using the same procedure as outlined for Pyrex glass No. 7740.

Copper to Glass Seals

These seals were developed by Housekeeper,[2] who found that under the proper conditions any type of glass may be sealed to copper. Pyrex with a coefficient of expansion of 32×10^{-7} compared to 166×10^{-7} per degree centigrade for copper can be sealed directly if care is taken in preparing the copper to be sealed.

Copper Tubing to Glass Tubing. These seals are frequently used in laboratories and are made three different ways, i.e., with the glass tubing sealed inside the copper tubing; with the glass sealed outside the copper tubing; or with the glass sealed both inside and outside the copper tubing. To assure the most satisfactory seals, regardless of the type made, oxygen-free, high-conductivity copper should be used.

All copper seals have one requirement in common, that is, that the copper tubing be fashioned to a fine taper or feather edge. Figure 2A gives the specifications to which the tubing should be machined when the glass is to be sealed inside the copper tubing. After the machining has been completed, the copper should be thoroughly cleaned with carbon tetrachloride to remove any grease left during the lathe operation. The final step in obtaining the required feather-thin edge is to dip the copper in a dilute solution of nitric acid, taking care to see that the acid does not create an uneven or scalloped edge. When the edge is satisfactory the copper should be thoroughly washed with distilled water to remove all trace of acid. It is then ready to be oxidized.

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[2] Housekeeper, W. G., *J. Am. Inst. Elec. Eng.*, 42, 954 (1933).

1. Oxidation of copper. The copper should be heated until the tapered section shows a bright interference color, indicating thin layers of oxide. It is next dipped in a saturated solution of borax and the excess solution shaken off. The copper is then heated to a bright red (900° C) and quenched in water. Fused cuprous oxide is formed by this process; the final color of the copper should be a bright scarlet with no trace of black cupric oxide. The borax flux may be eliminated but its use helps prevent overoxidation.

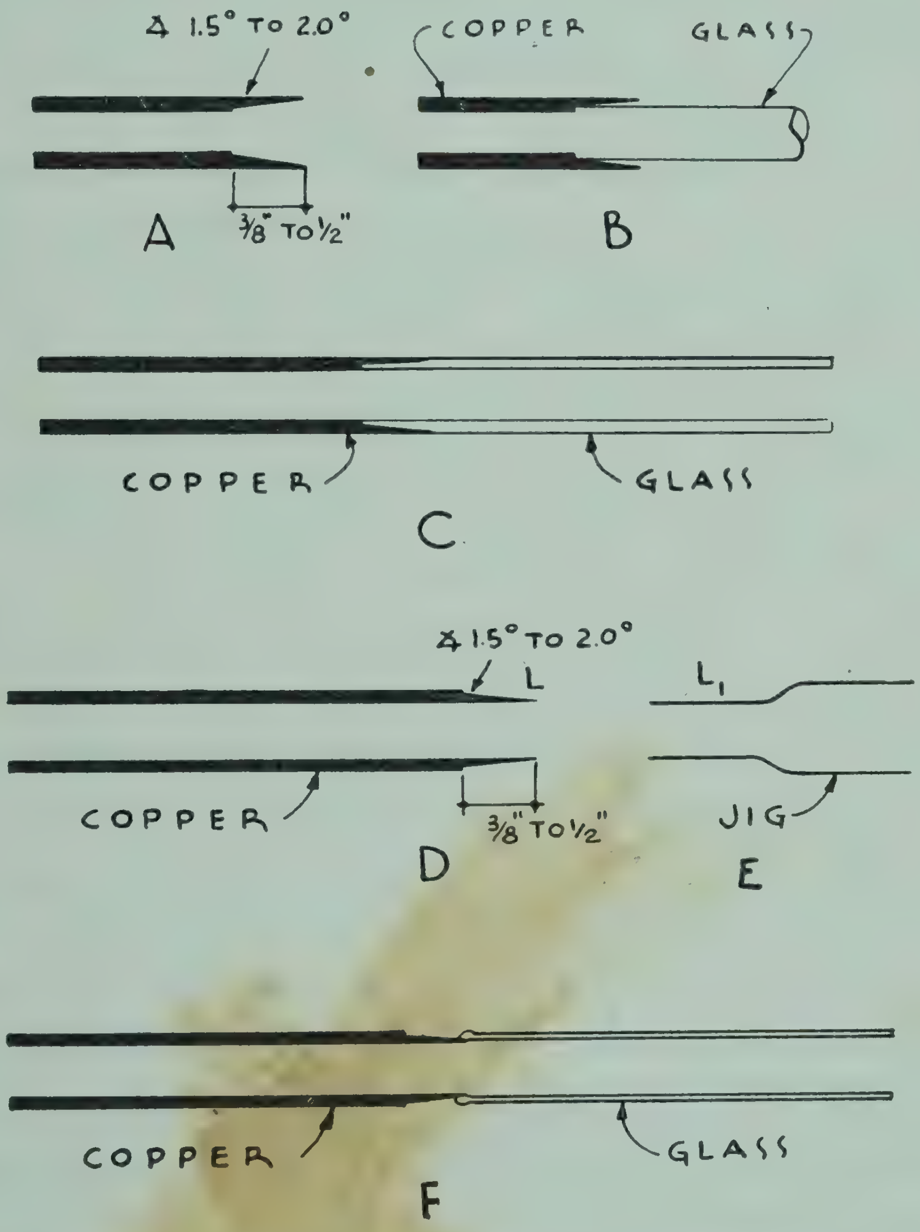
∡ 1.5° TO 2.0°
3/8" TO 1/2"
A
COPPER
GLASS
B
COPPER
GLASS
C
∡ 1.5° TO 2.0°
L
COPPER
3/8" TO 1/2"
D
L1
JIG
E
COPPER
GLASS
F

Figure 2. Steps for copper to glass seals.

2. Sealing glass inside the copper. A piece of glass is selected that will slide inside the copper tubing and butt up against the inner shoulder. The copper tubing should be supported in a handle to facilitate rotating during the sealing operation.

The copper and glass tubing are held and rotated as in making a straight glass to glass seal, except that a steady compressive pressure is maintained between the two hand. The burner flame is focused on the copper, and the heat conducted by the copper will soften the glass and allow it to expand out to the copper and form a bond. Blowing should be in a series of sharp hard puffs until the glass wets the copper and a seal is formed, after which a light but steady pressure should be maintained inside the glass. As the seal progresses, the flame should be brought gradually toward the thin edge of the copper. At no time, however, should the hot oxygen flame be focused directly on the thin edge. The seal is finished when the glass swells out to meet the copper as shown in Figure 2C. Care should be exercised to see that the glass does not expand back over the thin edge of the copper; should this happen the seal will probably crack on cooling. When properly made, copper to glass seals of this internal type are satisfactory from the standpoint of freedom from strain.

3. Sealing glass outside the copper. Figure 2D gives the specifications for machining copper tubing for outside glass seals. A jig should be made (Figure 2E) for inserting in each size of copper tubing to be turned down, and in each case the outside diameter of the jig should fit the inside diameter of the copper tubing. The length (L^1) is not critical but is must be longer than (L) of the copper taper. In machining the copper, the jig is inserted, the lathe adjusted for cutting a 1½° to 2° taper, and the copper cut away until the thin edge is obtained. After being thoroughly cleaned with carbon tetrachloride to remove any grease or oil, the machined copper is dipped in dilute nitric acid as described for internal glass to copper seals. The copper should then be washed with distilled water, dried, and oxidized as previously described.

A piece of glass is selected which will just slide over the thin section of copper tubing. The copper tubing is supported in a handle to facilitate rotating the work during the sealing operation. Both the copper tubing and glass are held and rotated as in making an internal copper to glass seal. A light compressive pressure is maintained between the two hands, and a slight vacuum is maintained inside the glass by sucking on the blow tube, until the glass wets the copper and the seal is formed. The burner flame is focused on the glass, and at no time should the flame be directed on the bare copper. A finished seal is shown in Figure 2F. The completed copper to glass seal will have a layer of cuprous oxide between the glass and the metal which will give it a crimson color. Black Seals should not be used.

4. Combination internal and external glass seal. To make these seals, the following steps are used. The copper tubing is machined as shown in Figure 3A. A Pyrex glass tube with an outside diameter that fits snugly into the copper tube is selected and flared, as shown in Figure 3B. The copper tube is mounted in a suitable holder for ease in rotating and oxidized by heating until it is hot. Borax may be used as a flux during this oxidizing heat. The flared glass is then pushed into the hot copper and tooled both inside and outside until it is fused with the copper (Figure 3C). A piece of glass tubing may now be sealed on as in making a straight butt seal (Figure 3D).

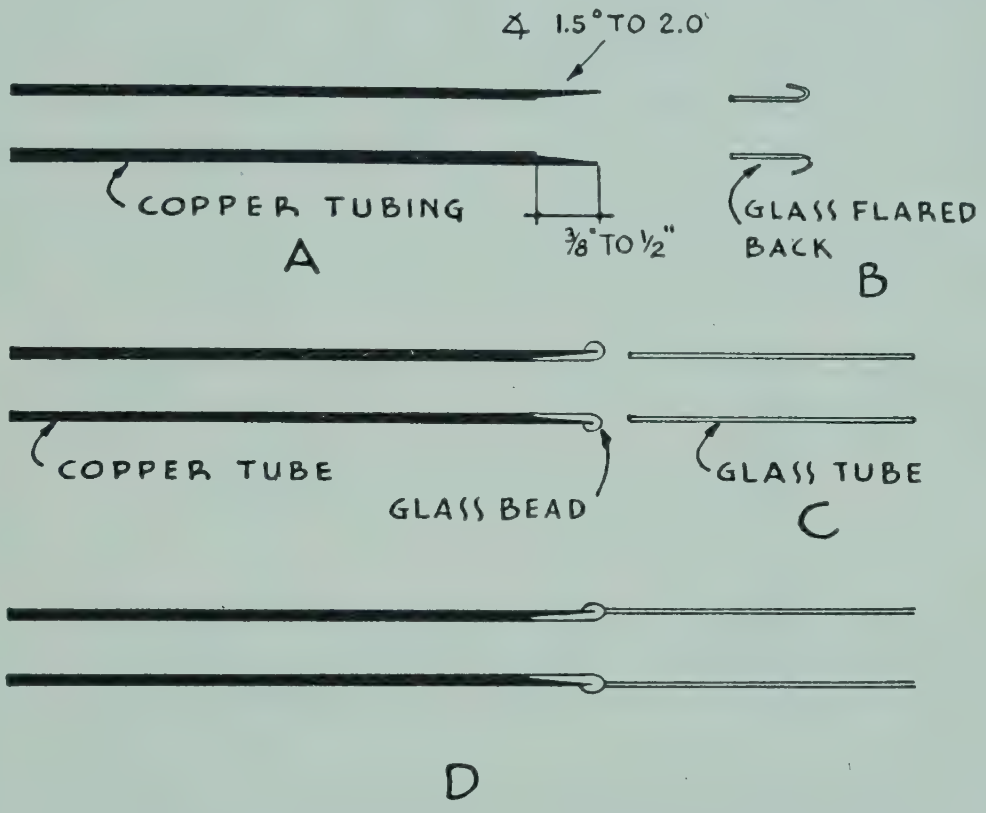

Figure 3. Combination external and internal copper-glass seal.

5. Preparing copper tubing for glass seals by rolling. In place of machining copper tubing to a thin feather edge it is

possible to prepare it by what could be called the "rolling method".

In this method the copper tubing is placed in a machine lathe and rolled to the desired thickness by means of a roller or tool supported in the same fashion as a knurling tool. A close-fitting mandrel inside the copper tubing provides the surface to roll against.

Copper tubing of not over 0.025 in. wall thickness may be rolled directly, but when heavier stock is used the wall thickness should be rolled to not over 0.030 in. before the actual seal roll is begun. The copper tubing should be annealed by being heated to about 600° C, and quenched in water before rolling is begun. If the copper has been properly annealed no wax or lubricant will be necessary during the rolling. Since it is not possible to roll tubing with the degree of accuracy obtained by machining it, the following table of measurements may be used to check the accuracy of the roll.

Thickness of copper, inches	Distance from edge, inches
0.030	5/8
0.028	1/2
0.017	3/8
0.011	1/4
0.006	1/8
0.0025	1/16
0.001	0

A ball and anvil micrometer should be used to measure the thickness of the copper and great care should be observed in measuring the thin portion to avoid damaging the edge.

After the rolling the copper should be given the carbon tetrachloride wash and the dilute nitric acid dipping if the edge is not 0.001 in. or less.

(*Copper Disc Seals.* These seals are used extensively in ultra-high-frequency techniques. A disc seal may also be convenient for sealing a heavy metal electrode into a glass vacuum system. The copper disc should be about 0.007 to 0.010 in. thick with an overlap of ⅜ to ½ in. beyond the diameter of the glass tube to be used. The first step is to borate the copper, as described previously for tube seals. It is advisable to cut the glass and grind it with square ends and fire-polish. The polished surface will produce a seal free from air bubbles. The two glass tubes are placed in a lathe with slight pressure so that the copper disc is held between them. If an induction furnace is available, the seal can be made easily and rotating is unnecessary. If a flame is used, it should be played on the edge of the disc, and directed tangentially rather than toward the center of the disc. Different stages in the making of this seal are shown in Figure 4. By leaving a hole through the disc, leads can be inserted and soldered in place.

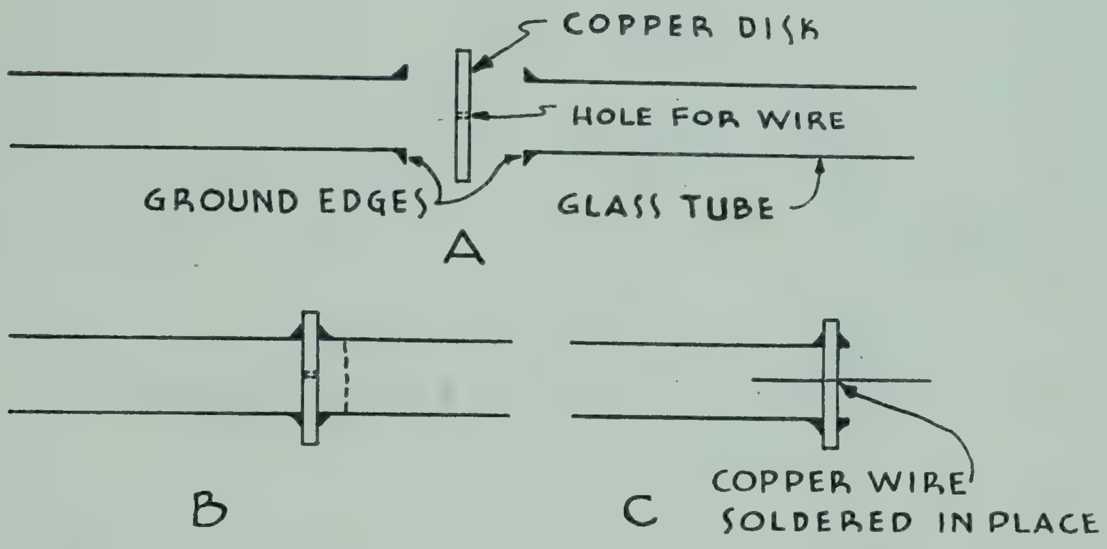

Figure 4. Copper disc to glass.

Copper Wire to Glass. Seals can be made if the copper wire is flattened until the section to be sealed into the glass is not over 0.002 in. thick, (see Figure 5A). The preliminary sleeve beading method used for other wire to glass seals is not used because of the danger of overoxidation of the copper. The glass is prepared by heating and drawing out a length of glass of the same wall thickness as the original tube, but of smaller diameter. The length of this small section should be over ½ in. This is cut from the larger section and if the inside diameter is too large for the copper wire, the section is reheated until the opening is the proper diameter for the flattened copper wire to slide in. The flattened copper wire is prepared by heating, cooling, dipping in borax solution, reheating and quenching in water, the same as for copper tubing. The seal is made by inserting the copper wire into the glass, heating the glass (without burning the copper in two), and making a press seal with a pair of tweezers.

Copper Ribbon to Glass Seals. These can be made if the copper ribbon is filed as shown in Figure 5B. The treatment is the same as for copper wire. Any part of the copper which is to be sealed to glass must be free from grease, finger marks, and dust.

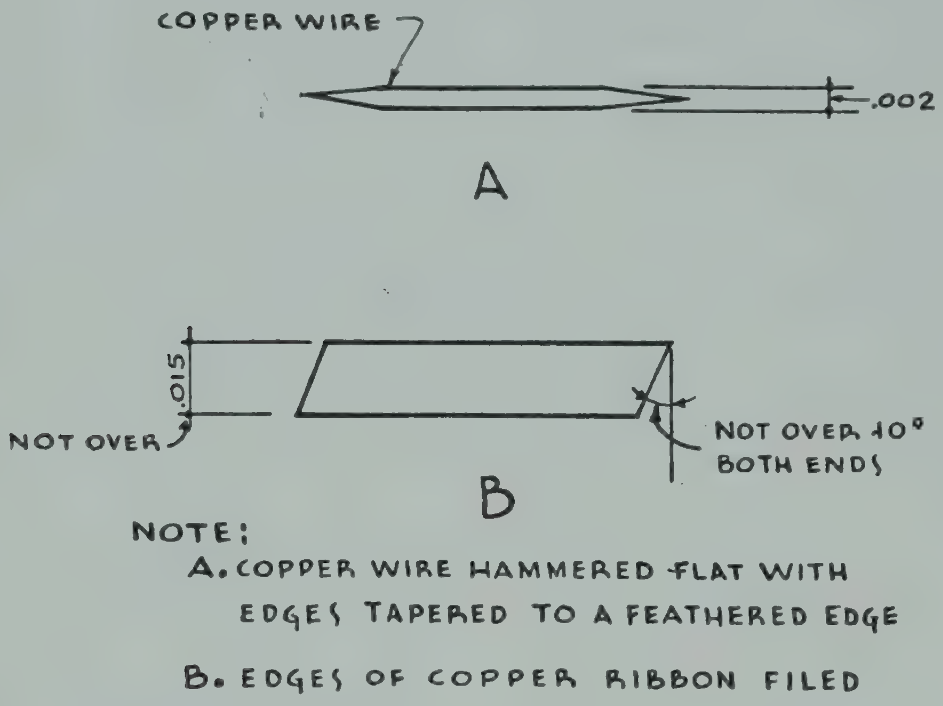
COPPER WIRE
.002
A
.015
NOT OVER
NOT OVER 10°
BOTH ENDS
B
NOTE:
A. COPPER WIRE HAMMERED FLAT WITH
EDGES TAPERED TO A FEATHERED EDGE
B. EDGES OF COPPER RIBBON FILED

Figure 5. Preparation of copper wire and ribbon for sealing to glass.

Cleaning Completed Copper to Glass Seals. After copper to glass seals are completed and annealed, they should be immersed in an acid bath for 10 second to clean the copper. The composition by weight of this bath is as follows:

Sulfuric acid	58.80 %
Nitric acid	7.20 %
Hydrochloric acid	0.25 %
Water	33.75 %

Platinum to Soft Glass Seals

These seals are made quite easily since soft glass and platinum have about the same coefficient of expansion. However, since lead glass wets the surface of the metal more easily than soda lime glass, it is recommended for use whenever possible. Platinum wire should be beaded before the seal is attempted. Figure 6 gives the steps in making several types of platinum to glass seals.

A close-fitting bead, 2 mm. I.D. × 10 mm. long, is slipped over the wire and heating is started at one end and continued progressively toward the other end; care is taken to avoid trapping air between the glass and the metal. After the wire is beaded, it must be kept hot while the opening in the tube is prepared so as to avoid the danger of the bead's cracking during reheating for the final seal.

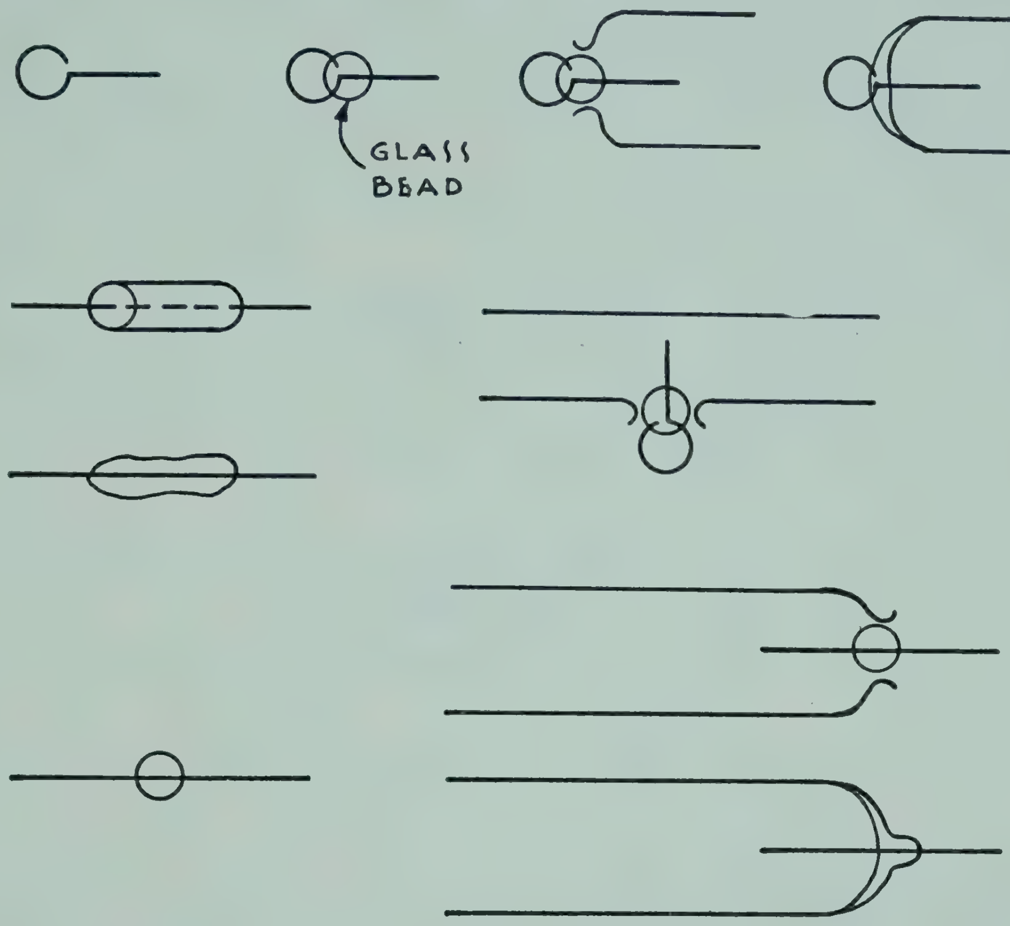

Figure 6. Steps in platinum to soft glass seals.

Platinum to Pyrex Seals

These seals can be made by two methods.

1. A very light-walled platinum tube is used. The Pyrex is fused about the outside of the tube for a distance of 5 to 15 mm. If the platinum is required to carry current, its capacity can be increased by passing a platinum wire through the tube and welding it at one end (Figure 7).

2. Fine wire is flattened and the edges filed to a sharp V shape. A glass tube is shrunk to a diameter only slightly larger

than that of the flattened wire and the glass is pressed with tweezers to assure good contact with the metals.

Platinum having a coefficient of thermal expansion of 90×10^{-7} must be thin enough when sealed in Pyrex that it will yield with the glass rather than tear away from it. Such a seal will be liquid-tight but is not intended for use with vacuum.

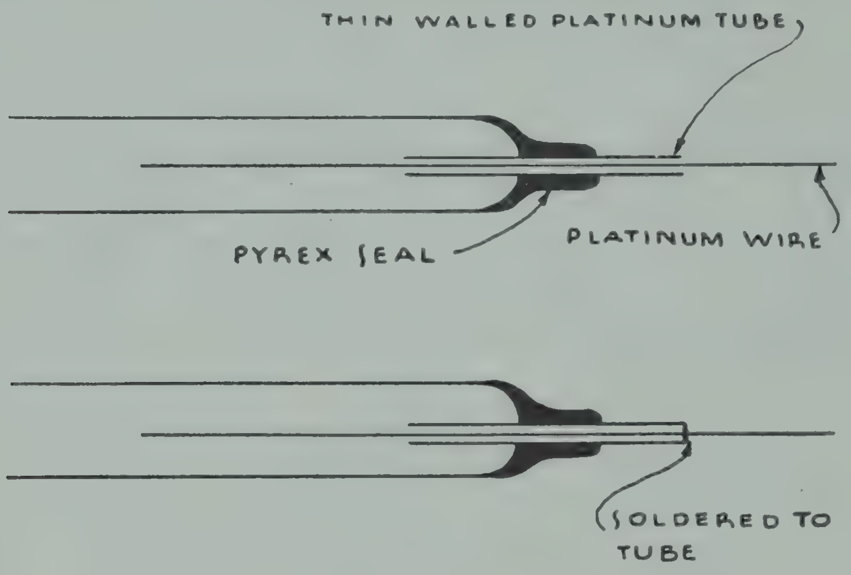

Figure 7. Platinum to Pyrex seals.

Kovar to Glass Seals

Kovar is a cobalt-nickel-iron alloy which has been used commercially since 1934 and was developed specifically for sealing to a hard glass. These seals are very sturdy when properly made. Figure 8 shows the expansion of Kovar as compared to two commercial hard glasses. Very fine wires may be sealed directly to Pyrex glass and some heavier seals may be made to Nonex. Most Kovar seals, however, require special Corning glasses No. 705 A.J. and 704.

The cleaning and degassing of Kovar preparatory to sealing it to glass is one of the most important steps in producing strong hermetic seals. The surface of the Kovar in the area of the seal should be free from marks or scratches which extend from the sealing surface outside, although radial marks are not objectionable.

One distinct advantage of Kovar over copper for glass seals is that no fine feather edge is necessary, making for much greater mechanical strength. When an end seal is to be made on Kovar,

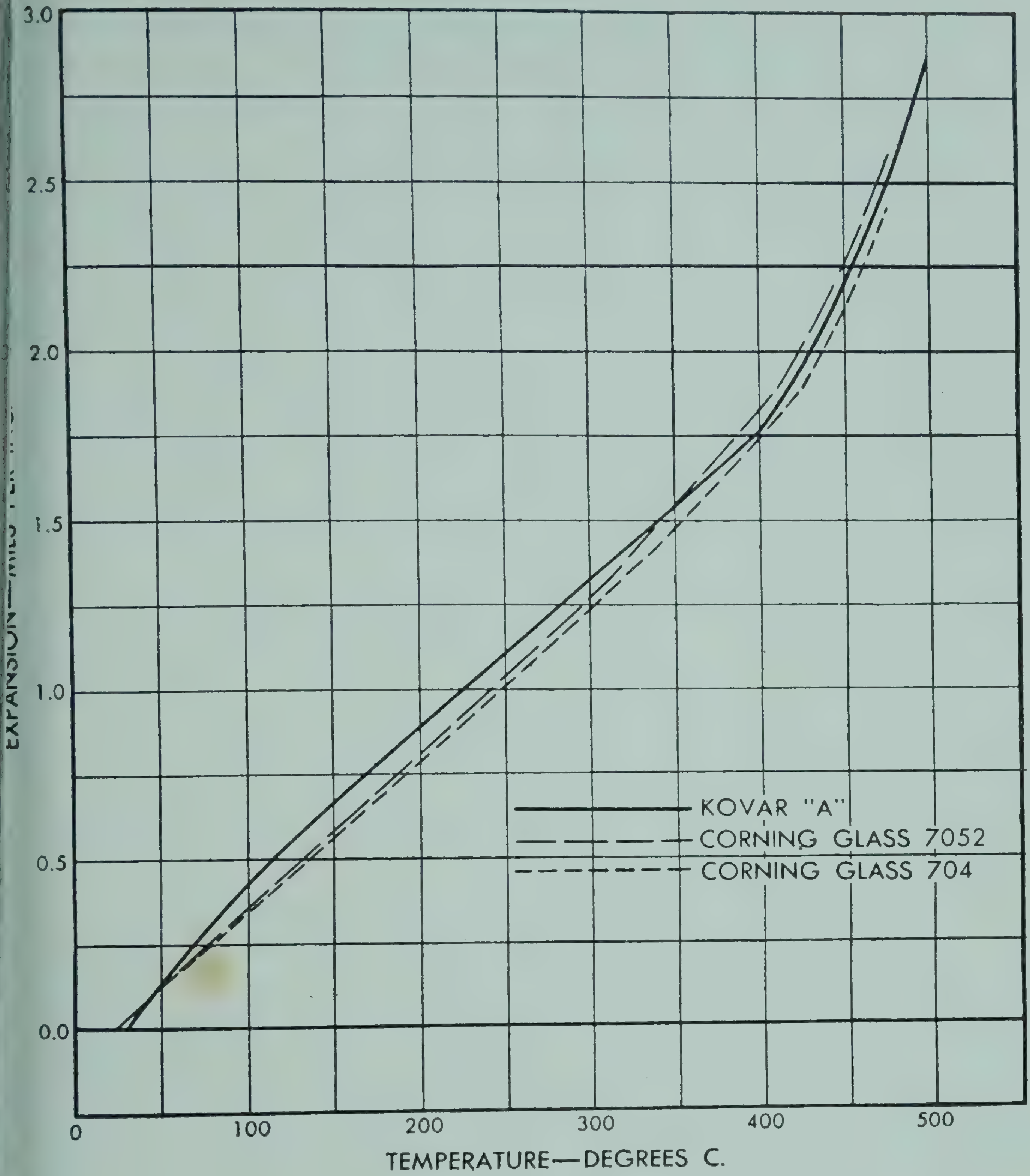

Figure 8. Expansion of Kovar to two glasses.

the sealing edge should be well rounded with all sharp corners removed. Deep scratches should be removed by turning the Kovar on a lathe. Small scratches may be removed by polishing with Aloxite cloth of #120 grit. A high or finished polish may be obtained with Aloxite #24002 #320 grit.

Machining. Kovar acts like stainless steel and may be readily machined at slow speeds with high-speed cutting tools and a lubricant such as lard oil.

Drawing. Kovar may be readily drawn. Stupakoff[3] recommends the following rule for drawing Kovar.

First draw	Max. reduction	40 %	
Second draw	Max. reduction	25 %	
		30 %	after reannealing
Third draw	Max. reduction	20 %	
		25 %	after reannealing

Annealing. Kovar is annealed when subjected to a temperature of 700° to 1000° C. for 1 hour. Annealing should be done in an atmosphere of hydrogen, although a furnace with a neutral atmosphere may be used, provided the hydrogen anneal is employed just before the glass sealing operation.

Degreasing. The metal must be absolutely clean if a hermetic seal to glass is to be made. Cleaning may be accomplished by vapor degreasing, and in this operation it is important that the cleaning fluid be free of residual greases. Any design which permits a flow of fresh cleaning vapors to wash the surfaces of the metal is satisfactory. An acetone wash after degreasing further assures cleanliness.

Degassing. After the cleaning and just prior to sealing, the Kovar should be furnace degassed and annealed in a hydrogen atmosphere made moist by bubbling hydrogen through water. When the Kovar is not free of occluded gases or carbonaceous matter, gas bubbles will appear in the finished seal. These bubbles reduce the mechanical strength of the seal and constitute possible

TABLE I. Degassing and Annealing Kovar

Temperature, °C.	Time	Cooling
800°–900°	2 hours	The Kovar should be cooled to 300° C. before being removed from the hydrogen atmosphere.
900°–1000°	30 minutes	Cooled to 300° C. before removal.
1100°	10 to 15 minutes	Cooled to 300° C. before removal.

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[3] Stupakoff Ceramic and Manufacturing Co., Latrobe, Pennsylvania.

vacuum leaks when the seal is used for vacuum work. Table I gives the time and temperature used for degassing Kovar. It is important that degassed Kovar parts be protected from grease and dust prior to sealing, and it is recommended that the seals be made as soon as possible after cleaning and degassing—not over a few hours at the most.

Extreme care should be taken in handling the degassed Kovar parts in making the seals. Touching the part to be sealed may cause bubbles in the finished seal, and such mishandling of the parts after degassing defeats the purpose of that operation.

Reoxidation. As in other glass to metal seals, the actual seal is made by the Kovar oxides dissolving into the glass. For this reason it is necessary to form an oxide film on the Kovar part prior to the sealing operation. This oxidation can be accomplished by rapidly heating the metal to over 650° C. in a slightly oxidizing atmosphere.

Kovar parts should not be heated in the reducing portion of a flame. When possible, uniform oxidation may be accomplished by the batch method in a furnace. In contrast to copper to glass seals, heavily oxidized Kovar produces a mechanically strong but porous seal, whereas underoxidized seals are more gastight but mechanically weaker. Correct oxidation results in a seal having a mouse-gray or brownish appearance, depending on the glass used. Kovar oxide is more readily soluble in Corning No. 7052 glass than in Corning No. 704 and for that reason seals made with No. 7052 are lighter in color than seals made with 704. Excessive oxidation may be removed by either prolonged heating or raising of the sealing temperatures. However, either procedure increases the danger of devitrification.

Sealing is basically wetting the oxide with glass in a plastic state. The design and size of the seal determine whether heating will be localized to the seal or spread over the work so as to prevent cracking of the glass apparatus. A slight pressure of the glass on the metal starts the sealing action and, when possible, some mechanical tooling of the glass is helpful during the sealing operation. Feathering of the glass along the surface

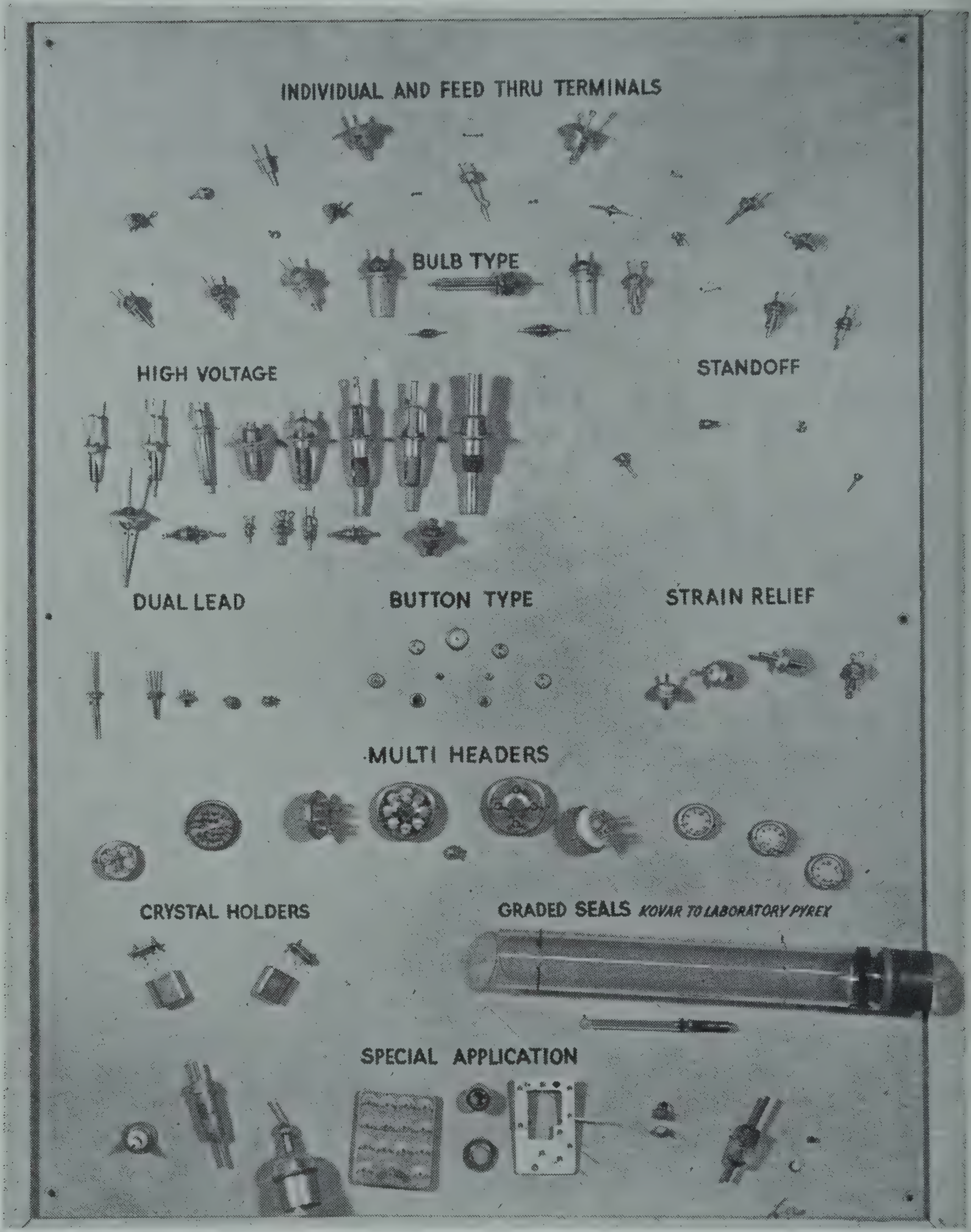
INDIVIDUAL AND FEED THRU TERMINALS
BULB TYPE
HIGH VOLTAGE
STANDOFF
DUAL LEAD
BUTTON TYPE
STRAIN RELIEF
MULTI HEADERS
CRYSTAL HOLDERS
GRADED SEALS KOVAR TO LABORATORY PYREX
SPECIAL APPLICATION

Figure 9. Some forms of commercial Kovar to glass seals.

of the metal should be avoided when maximum mechanical strength is desired. Figure 9 shows commercial seals of Kovar to glass.

Annealing Kovar to Glass Seals. Flame-annealing of Kovar to glass seals is often used. A carbon deposit from a yellow flame maintains a desirable cooling rate. Oven annealing is shown in Table II.

TABLE II. Annealing Kovar to Glass seals

From room temperature to annealing temperature of the glass	30 minutes
Held at annealing temperature of glass	20 minutes
Cooled from annealing temperature to strain point	1° C. per minute
Cooled from strain point to room temperature	7°–10° C. per minute

Electronic Sealing. High-frequency currents, ranging from 40,000 to 60,000 cycles per second, from an induction furnace can be used for Kovar to glass seals. In these seals it is imperative that the glass and metal parts be in contact, since the working temperature of the glass is attained by conduction from the Kovar. This method of sealing can be used very advantageously because heating is localized and glass deformity is limited to the point of metal contact.

Powdered Glass to Kovar Seals. Powdered glass may be used to make satisfactory glass to metal seals. In this operation the Kovar should be preoxidized in a furnace at 650° C. for about 6 minutes. A coarse flour of powdered glass and methanol is prepared. No binder other than methanol is necessary in preparing the flour, and the two are mixed in the proportions of 1 : 1 by volume. The powdered glass is sprayed on the Kovar and fused to the metal either by baking in an oven or by induction heating. After this fusion is complete, a glass window or a tube of glass may be sealed to the ring of fused glass by direct heat or induction heating.

Nickel-Iron to Glass Joints

An alloy of nickel and iron gives a coefficient of expansion remarkably similar to that of some glass at low temperatures. It has been found that with these alloys the coefficient of expansion varied as the proportions varied. The 30 % alloy is Invar. Its expansion is very sensitive to the composition and may, on occasion, be negative. With Invar tubing, glass is usually sealed on the outside of the tube. It is advantageous to thin the alloy near the glass joint but not to the extreme thinness necessary for copper joints.

Nickel-Iron Alloys

Percentage of Nickel	Expansion Coefficient per ° C. at Room Temperature
10	13×10^{-6}
20	19.5×10^{-6}
30	12.0×10^{-6}
36	0.9×10^{-6}
40	6.0×10^{-6}
50	9.7×10^{-6}
80	12.5×10^{-6}

Dumet to Glass Seals

Dumet wire is a 40 % nickel, 60 % iron alloy which is coated with a substantial layer of copper amounting to 20 % to 25 % of the cross section. The overall expansion of the alloy is of the right order for glass seals, and the copper provides the necessary conductivity as well as the basis for the glass seals. The upper limit for a safe seal with Dumet is wire 1 mm. in diameter. In the case of some commercial lamps, Dumet wire is used not only in the seal but also for supporting the tungsten filament. It can be used for inserting copper electrodes into glass by flaring a cup or thimble of the alloy to which the copper is brazed or soldered.

Chrome-Iron to Glass

A chrome-iron alloy consisting of 75 % iron and 25 % chrome, having an average expansion coefficient of 10×10^{-6}, can be wet by glass and thus permit a direct glass to metal joint to be made without the necessity of the intermediate copper plating. The alloy is machined down as shown in Figure 10. The alloy is next oxidized slightly in an oxidizing flame and a thread of glass melted onto the edge of the metal at the proposed line of junction. The joint is completed by fusing the glass tube to the ring of glass already sealed to the metal. The overlap should not be more than 1 to 7 mm. wide. This alloy is slightly harder than mild steel and has a low heat conductivity. It can be heated without injury up to 1000° C., at which temperature hydrogen will diffuse through it.

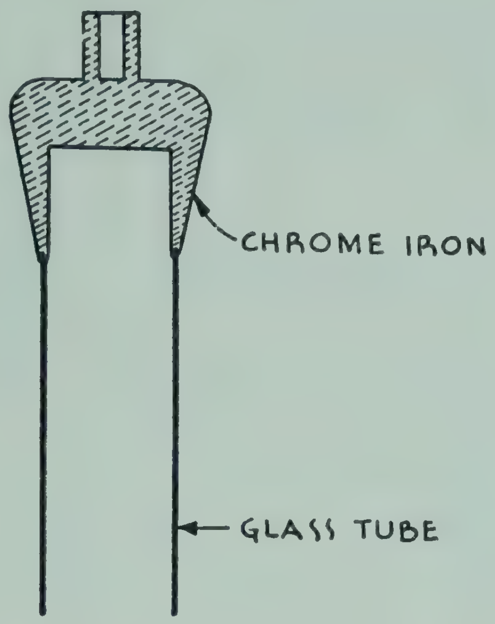

Figure 10. Chrome-iron to glass.

Platinized Glass to Metal Joints

It is still convenient at times to use one of the oldest methods of making glass to metal joints, namely, through the intermediary of a platinum deposit. A colloidal solution of platinum in lavender or other essential oil was developed by McKelvy and Taylor.[4] The solution is painted on the glass and heated in a Bunsen burner, thus depositing a coating of platinum. This is then copper-plated and the joint to the metal tube completed by soldering. Cailletet[5] found that such a joint would withstand an internal pressure of 300 atmospheres. It has been found advantageous to roughen the glass before platinizing and then to copper and silver plate alternately over the joint. This method can be used equally well with clear silica. Meyers[6] found that either a platinized or unplatinized glass tube, warmed and dipped quickly into a bath of molten lead, will have a thin coating of lead to which metal can be soldered. The lead should be at a temperature only slightly exceeding its melting point and the temperature of the glass should be halfway between that and room temperature.

[4] McKelvy, E. C., and Taylor, C. S., *J. Am. Chem. Soc.*, 1364 (1920).
[5] Cailletet Seances, *Soc. Franc. Phys.*, p. 242 (1890).
[6] Meyers, C. H., *J. Am. Chem. Soc.*, **45**, 2136 (1923).

By using these lead joints, a seal may be made directly between glass tubes and metal tubes previously tinned. The glass should fit snugly for 15 to 20 mm. within the metal tube, although with care satisfactory seals may be made where the glass does not fit the metal tightly. In either case, however, the efficiency of the joint is increased by first platinizing the glass.

Copper to Glass by Electroplating

A method of making glass to metal joints by electroplating has been described by Dundon.[7] The first step in making seals by his method is the platinizing of the glass. This platinized section is then copper-plated and the copper plating polished to form a smooth sliding fit inside the copper tube to which it is to be sealed.

Copper is then deposited by electroplating until a smooth copper coating covers both pieces, uniting them completely. During the plating it will be necessary to remove the pieces occasionally and file the deposit at the joint to obtain a perfectly smooth joint.

Metal to Silica Seals

For these seals clear silica should be used since the translucent silica is apt to be porous. Clean molten lead possesses the property of wetting silica and can be used for sealing tungsten or molybdenum electrodes into silica. Sands[8] developed the following method for introducing metal through silica. A silica tube is shaped as shown in Figure 11. A molybdenum wire is placed loosely at A and a piece of lead higher in the tube. The tube is exhausted by a mechanical pump, and the glass is softened and pinched or collapsed onto the molybdenum wire. The lead is then melted and allowed to filter into space E which also has been heated. The upper end of the tube C is opened to the air while the lead is still molten so that the atmospheric pressure forces it well against the interior wall of the silica reservoir. The tube is then cut at E and a tinned lead-in wire is soldered in place.

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[7] Dundon, M. L., *J. Am. Chem. Soc.* **45**, 716 (1923).

[8] Sands, H. J. S., *Proc. Phys. Soc.* (*London*), **26**, 127 (1914).

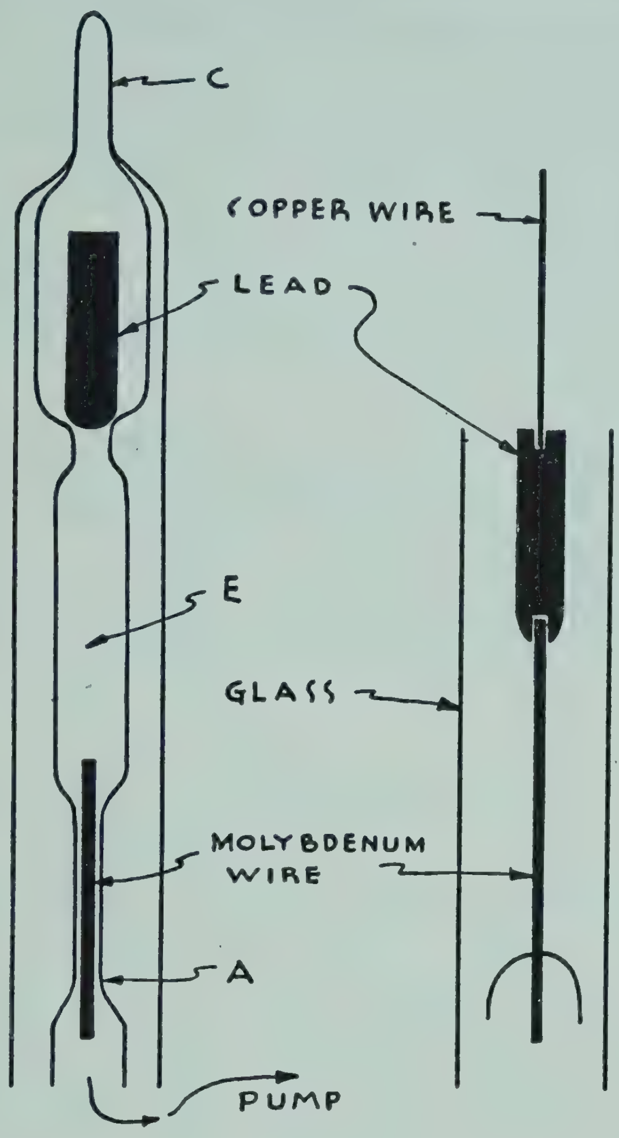

Figure 11. Molybdenum wire-silica seals.

Invar to Quartz

A second metal to silica seal involves the use of Invar and quartz. The expansion of the two materials is sufficiently close over a range of 0 to 200° C. to permit this seal to be made.

The seal is prepared by depositing a thin layer of platinum from a colloidal solution of platinum and lavender oil. This is heated in a Bunsen burner. The resulting platinum layer is built up by electroplating copper and the joint completed with tin solder.

Molybdenum Foil to Quartz

Satisfactory seals have been made with molybdenum foil and silica. Figure 12 shows the various steps of the seal.

A strip of molybdenum foil, 0.0005 in. thick and about 3/8 in. long, is spot-welded at each end to tungsten or molybdenum wires. The wire is placed at the sealed end of the silica tube, which is being exhausted continuously by a mechanical pump. The tube is then collapsed along the length of the molybdenum strip and slightly past the two spot welds. In spite of the greater coefficient of expansion of the metal, there is sufficient adhesion between the foil and the silica to give a vacuum-tight seal. Although the seal is not vacuum-tight at the point of the spot welds, this gives greater mechanical strength. A 4 mm. wide molybdenum strip will carry continuously up to 10 amp.

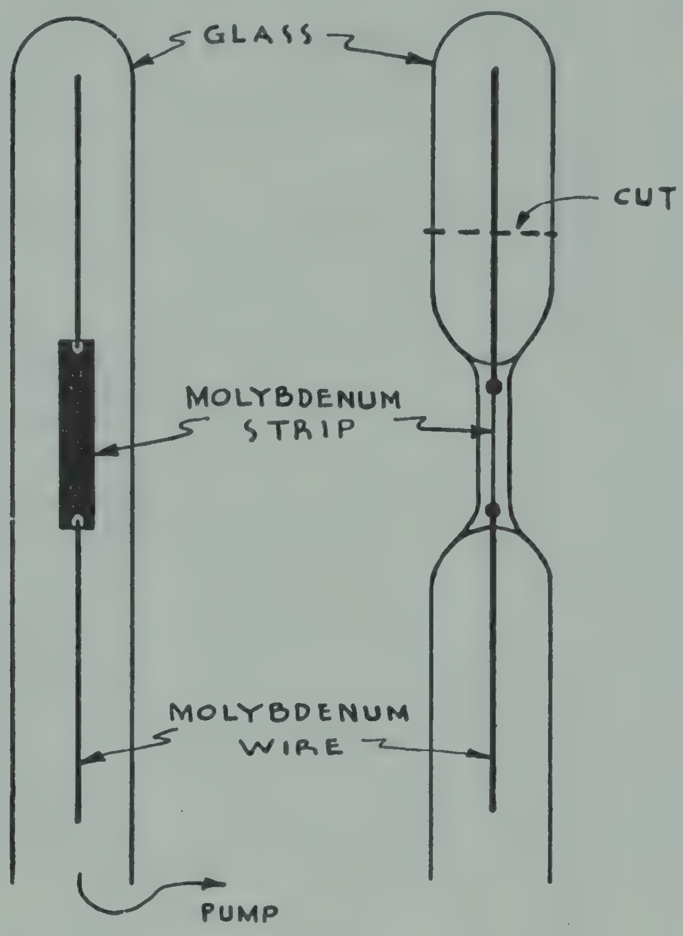

Figure 12. Molybdenum foil to silica seals.

Wrung Joints

These are produced by the adherence of optically worked surfaces, either glass or metal, and are capable of supporting a moderate vacuum. It is possible that these joints can be used as a means of securing observation windows where the use of cement is inadvisable.

Unfortunately, these joints are mechanically weak and liable to failure when subjected to sudden temperature changes. However, Parker and Palladay[9] have shown that two surfaces of similar glass in optical contact may be united by carefully controlled heating at temperatures much below their melting point. A joint made in this way is vacuum-tight and capable of withstanding reasonable temperature changes and mechanical shock. Within limitations, different kinds of glass may be sealed by this method.

CERAMIC TO METAL

Ceramic to metal joints are made by bonding a coating of metal to the ceramic material and soldering the metal member to it.

Hard Solder to Ceramic Joints

Greater care must be exercised in making hard solder to ceramic joints than when soft solder is used, because of the higher temperature at which the joint is made. However, the greater mechanical strength of the hard solder joint over the soft solder joint should be considered.

It has been suggested that metals with a slightly higher expansion than the ceramic be used for external seals so that the metal grips the ceramic, while for internal seals a metal with a lower expansion should be used.

A layer of metal may be fused to the ceramic by applying a coating of powdered metal or its oxide to the ceramic and firing it to a temperature of 1200° to 1300° C. in an atmosphere of dry hydrogen. The metal powder may be mixed with a heavy viscous liquid such as liquid paraffin or a 5 % solution of celluloid in amyl acetate. An even more adhering coating may be obtained by adding between 10 % and 20 % by weight of finely powdered glass. It is very important, of course, that the powdered glass used have the same coefficient of expansion as the ceramic.

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[9] Parker, R. S., and Palladay, A. J., *Trans. Faraday Soc.*, **12**, 305 (1916).

According to Partridge[10] layers of iron, nickel, and molybdenum have been bonded to the ceramics this way. Soldering directly to a molybdenum layer is difficult, but a coating of nickel deposited either by electroplating or burning on will correct this difficulty. It will be found advantageous also to plate the metal member with nickel. The soldering may then be carried out quite easily with silver for the sealing metal. Actual soldering should be done in an atmosphere of hydrogen at 1050° C.

Nickel may be burned on the ceramic by the following method.

A suspension of finely powdered nickel in a 5 % solution of celluloid in amyl acetate is painted on the surface of the molybdenum, dried, and fired in an atmosphere of hydrogen at 1100° C. to 1200° C.

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[10] Partridge, J. H., *Glass-to-Metal Seals*. Robert Clay, London, 1949.

IX

GRADED SEALS

At times it becomes necessary to seal glasses of different compositions together, such as soft glass to Pyrex or Pyrex to silica. Because of the great difference in their coefficients of thermal expansion these glasses cannot be sealed directly together, but by means of intermediate glasses, each differing successively by small increments in its coefficient of expansion, it is possible to make seals between the different glasses.

Pyrex to Soft Glass

Seals between Pyrex glass having a coefficient of expansion of 32×10^{-7} and soft glass with an expansion of 90×10^{-7} require six intermediate steps or glasses to make a satisfactory seal. It is possible to purchase a set of these intermediate glasses from Corning Glass Works. They are supplied in tubing form and by their use an excellent Pyrex to soft glass joint may readily be made. Figure 1 shows the order in which the glasses are sealed for a Pyrex to soft glass graded seal.

It is advisable when making Pyrex to soft glass seals to start with the Pyrex and work towards the soft glass. A gas-oxygen flame will be necessary for the initial seal between the Pyrex glass and the first intermediate glass, but a second gas-air torch should be burning and in position so that the change from one type of flame to the other can be made without delay. In addition to the two burners actually used in fusing the glasses together, a Bunsen burner should be available to keep the finished sections of the seal hot until the entire seal is completed and annealed.

The outside diameter of the set of intermediate glasses as purchased from Corning is about 12 mm. For most purposes these may be either drawn down or blown out to the desired size and the graded seal made as a series of straight or butt seals. When this method is used the length of each intermediate seal should be approximately equal to the diameter of the glass at the seal.

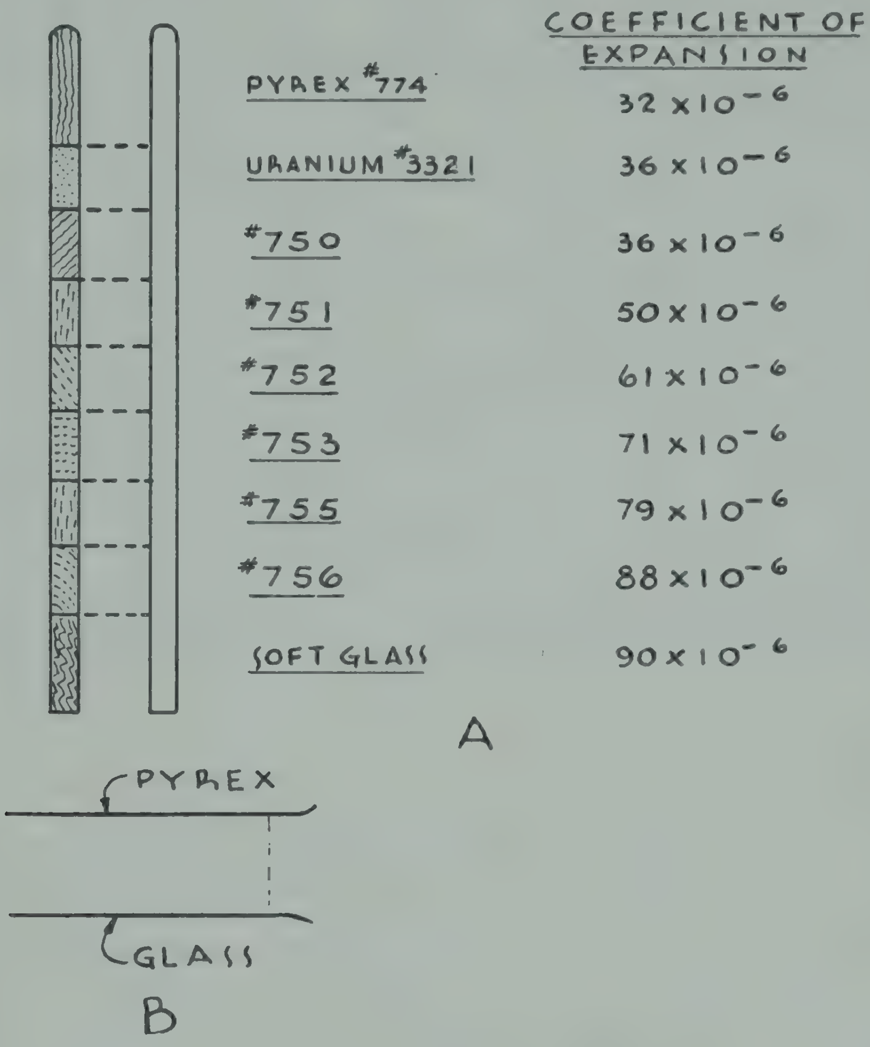
COEFFICIENT OF EXPANSION
PYREX #774
32 x 10⁻⁶
URANIUM #3321
36 x 10⁻⁶
#750
36 x 10⁻⁶
#751
50 x 10⁻⁶
#752
61 x 10⁻⁶
#753
71 x 10⁻⁶
#755
79 x 10⁻⁶
#756
88 x 10⁻⁶
SOFT GLASS
90 x 10⁻⁶
A
PYREX
GLASS
B

Figure 1. Pyrex to soft glass graded seal.

Because of the differences in working temperatures of Pyrex and the first intermediate grade of soft glass, sealing a section of uranium glass (No. 3321) to the Pyrex before beginning with the graded seal will prouce a better seal from the standpoint both of mechanical strength and appearance. A second method of making graded seals between Pyrex and soft glass is to draw a portion of each grade of glass (graded seal series) into a rod. For convenience the rods may be labeled 1 to 6 inclusive, starting with glass No. 750 as No. 1. The procedure for making the joint is as follows. A section of the Pyrex tubing is rotated in a flame until it begins to soften. At this point a uniform ring of glass No. 1 is quickly fused on the end of the Pyrex tube and blown out as shown in Figure 1B. This operation is repeated with each of the intermediate rods, with the soft glass finally sealed on after rod No. 6 has been applied. To avoid confusion in the application of the rods while making the seal, a board should be prepared for holding the rods. This board should have two rows of six holes each. The glass rods to be used should be placed in the front row in numerical order and then placed in the back row after they have been used.)

Pyrex to Nonex

(The expansion coefficients of Pyrex and Nonex glasses are similar enough to make direct seals between the two glasses. Because of the difference in working temperatures, however, seals between the two are more easily made if an intermediate step of uranium glass (3321) is used. The best procedure is to seal the uranium glass to the Pyrex; then, with the burner adjusted to produce a highly oxidizing flame, the Nonex glass is sealed to the uranium.)

Pyrex to Corning No. 705AJ (Kovar)

(When a seal between Pyrex and Kovar is required, a graded seal must be used. This graded seal consists of the following Corning glasses: Pyrex, uranium, Nonex, 705AJ or 705BA. Either of the last two glasses may be sealed to the Kovar. The procedure for making the graded seal is the same as that previously outlined for other graded seals.)

Pyrex to Silica

Commercial graded seals between Pyrex and silica are available from many supply houses, and they can be obtained in several diameters. Some suppliers will make graded seals of any specified size on order.(Graded seals are made by sealing Corning glasses No. 790-723-720-724-7740, starting with silica and working through the series in the order given.

Graded seals may be made by preparing the glasses for the intermediate steps as follows) (percentages are by weight):

Glass 1	92 %	Silica
	8 %	Boric acid
Glass 2	82 %	Silica
	18 %	Boric acid
Glass 3	72 %	Silica
	28 %	Boric acid

In preparing the silica it should be heated first to a red heat, held at this for 1 hour, then cooled and weighed. The silica should then be ground in a mortar and pestle and screened through a 150-mesh sieve. The boric acid is dehydrated and mixed with the silica in the proper proportion and screened through a 150-mesh sieve. Each separate step is then heated to the fusing temperature in an oven. This may be done in a nickel or glazed porcelain cup and the mixture poured on a nickel sheet for cooling. The fusing leaves the glass full of air bubbles which may be removed by drawing the glass into a thin rod by heating it in a gas-oxygen flame; care should be taken not to overheat the glass in the process. Protracted heating will remove most of the bubbles and produce a rod of clear glass. The actual seal between quartz and Pyrex may be made by using these rods and following the procedure previously described in the section on sealing soft glass to Pyrex with rods made of the intermediate glasses. If Vycox glass (Corning No. 790) is available it may be used as a fourth intermediate step or seal between silica and glass No. 1 (see Figure 2).

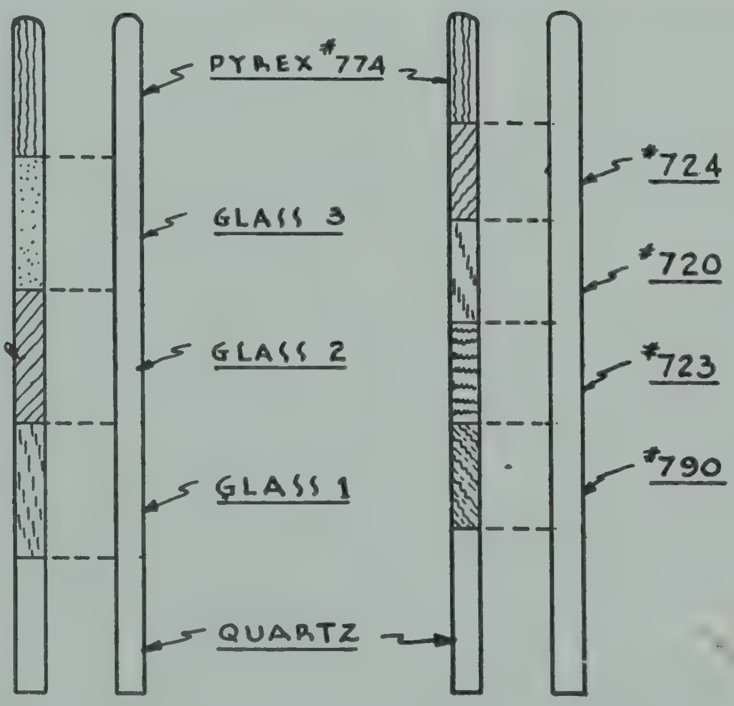

Figure 2. Pyrex to quartz graded seal.

Silica to Pyrex graded seals involving seven intermediate steps may also be made by grinding silica and Pyrex glass and mixing batches with the proportions of 4/1; 3/1; 2/1; 1/1; 1/2; 1/3; and 1/4. These batches are fused and drawn into rods following the procedure previously described.

MISCELLANEOUS SEALS

Quartz Windows to Pyrex Tubes

In sealing quartz windows to the end of a Pyrex tube, a piece of tubing the desired size is selected and the ends ground flat at a 90° angle with respect to the sides of the tubing. The edges of this squared end are then beveled to a taper. Bensen[1] found that the best angle for this taper was about 75° and grinding is done to such a depth that the flat edge is reduced to about half its width. See Figure 3A. When the taper is finished it should be fire-polished to present a diffused glossy appearance. Tapers which are not fire-polished show considerable strain and

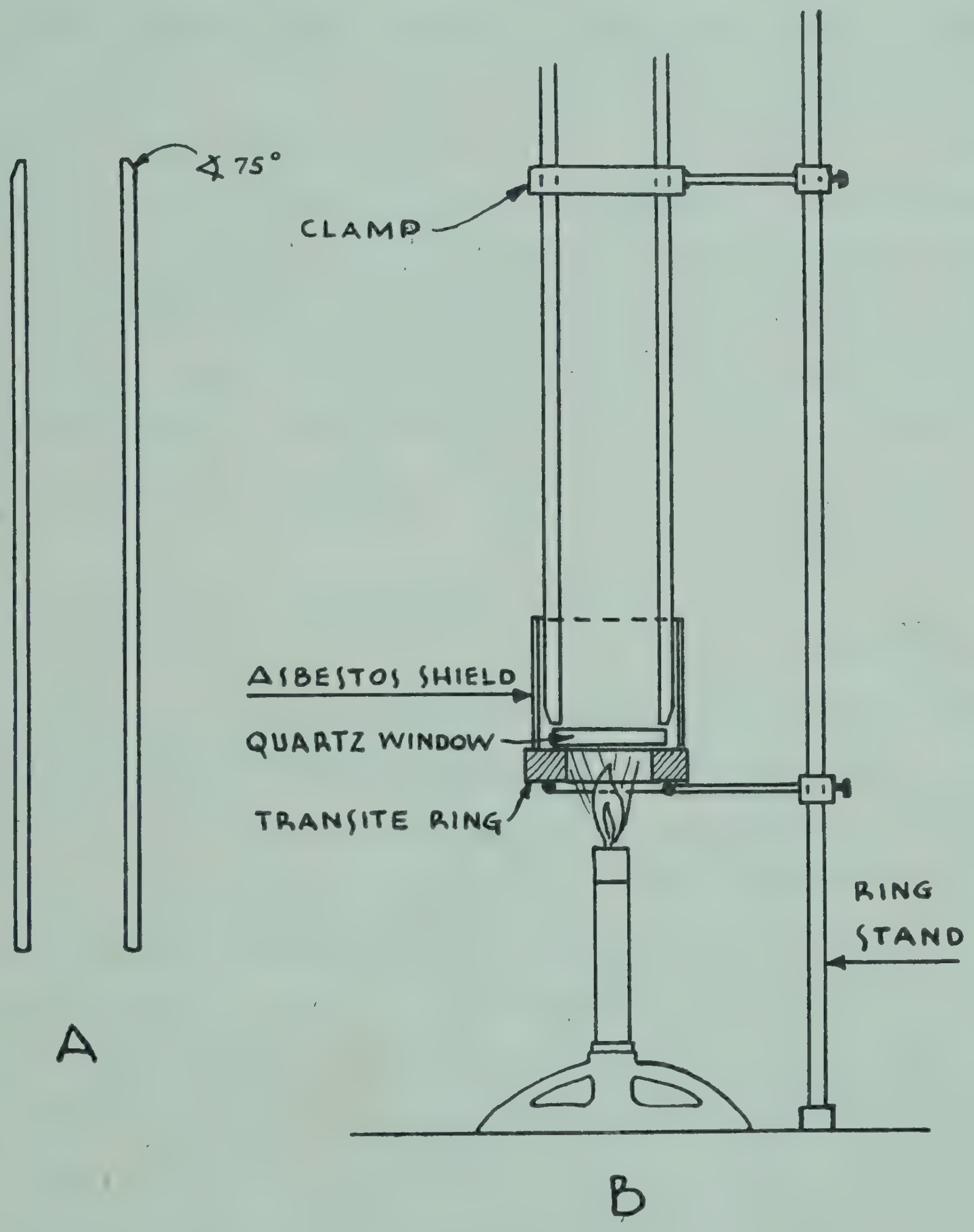

Figure 3. Quartz window to Pyrex tube.

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[1] Bensen, S. W., *Rev. Sci. Instr.*, **13**, 267 (1942).

generally crack. The ground surfaces are prepared by burning on three separate coats of platinizing solution. The quartz window should also have a thin ring of platinizing solution burned on around the edges. This may be obtained by resting the tubing on the window, painting both pieces with the platinizing solution, and flaming them. Each piece should be flamed separately to prevent the platinizing solution from forming a band. The tube is then coated with a 1 mm. coating of silver chloride by being repeatedly heated and dipped in a bath of molten silver chloride. When the proper coating has been obtained, both the tube and window are supported in such a way that the seal may be heated from below (Figure 3B). A piece of asbestos paper tape should be loosely wrapped around the tube to prevent stray drafts of air from cooling the seal too quickly, but it must be loose enough to allow it to be raised occasionally to observe the seal. When all is in readiness the quartz window is heated from below, causing the silver chloride to melt and flow smoothly around the seal. The finished seal should be annealed immediately and allowed to cool slowly.

Mica to Glass

Dorel[2] found that mica could be sealed to glass or metal by means of a low-melting lead borosilicate glass developed by Gallup.[3] This glass has a melting point of 450° C. and a coefficient of expansion of 98×10^{-6} in a temperature range of 0 to 375° C. For large seals the end of glass tube is cut and ground square to the axis of the tube, and the mica cut large enough to overlap the glass slightly. The special low-melting glass is finely ground and mixed into a paste with water. The paste is painted on the edge and end of the tube and the mica pressed into position. Some additional ground-glass paste is painted at the juncture between the mica and glass, and some ground-glass paste is painted over the edges of the mica to prevent its splitting later. The assembled parts are then placed in an oven and baked for 10 minutes at 600° C. The oven is then cooled slowly to room temperature before removing the seal. The cooling process should take at least 1 hour.

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[2] Dorel, J. S., Jr., *Rev. Sci. Instr.*, **13**, 266 (1942).

[3] Gallup, J., R.C.A. Manufacturing Co., Inc., Harrison, N.J.

Both Corning glass No. 8 (soda lime) and No. 12 (soda, potash, and lime) can be sealed to mica with this special powdered glass process. In addition, cold rolled steel may be sealed to mica by means of this same technique.

Ceramic to Glass

Most ceramic substances possess nearly uniform coefficient expansion characteristics from room temperature to temperatures

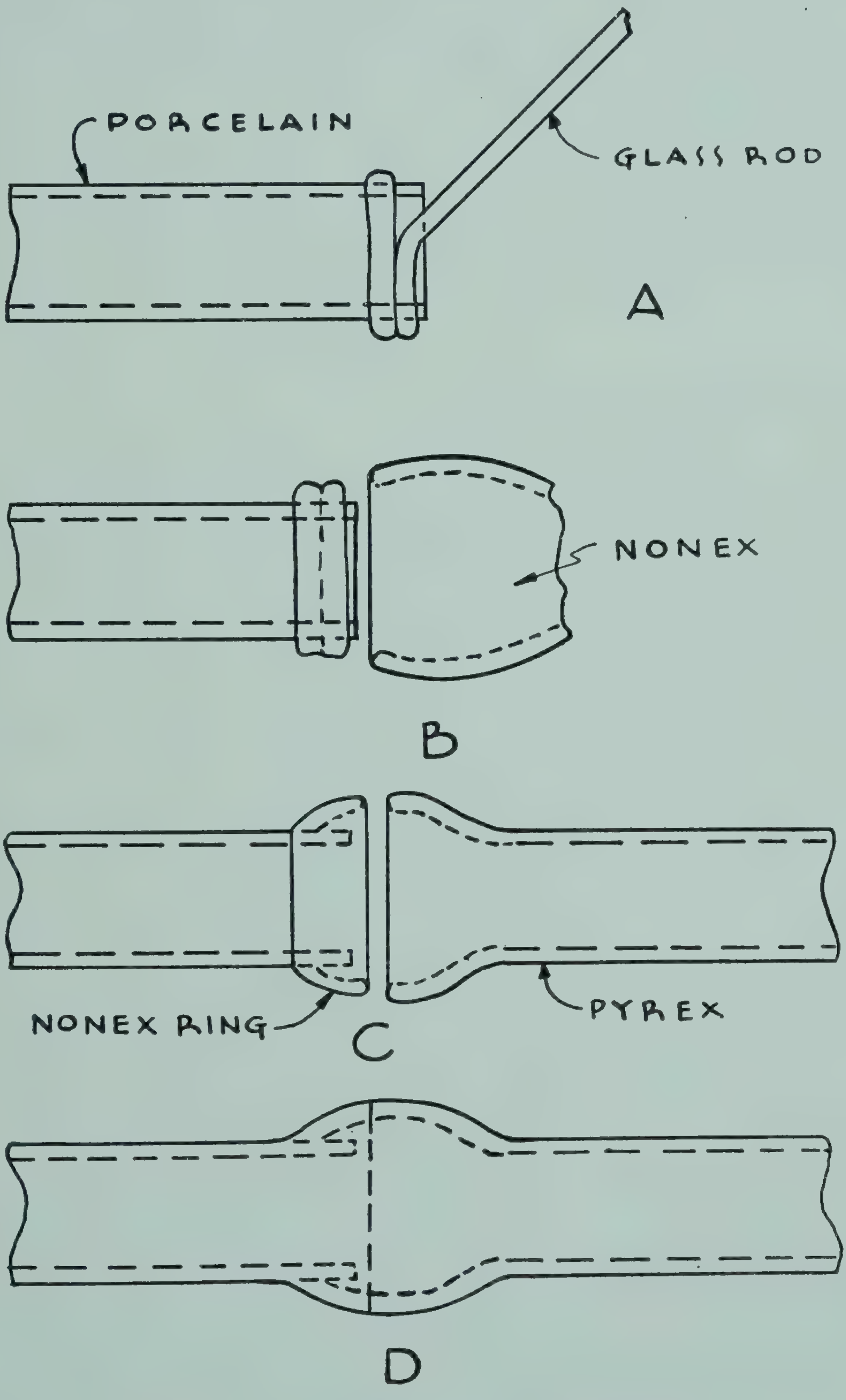

Figure 4. Ceramic to glass seal.

exceeding the annealing temperature of glass. For this reason the principles underlying glass to metal seals may also be applied to ceramic to glass seals, provided the expansion of the ceramic is known and a glass is used which will match it. Since commercial ceramics vary widely in their coefficients of expansion, the statement that a particular glass will seal to porcelain is of little use unless the composition and coefficient of expansion of the porcelain is known. Figure 4 shows the steps used in making a glass-to-ceramic seal. To obtain a strong seal, it is important that the glass does not flow over the edge of the porcelain, see Figure 4D.

Of prime importance in order to make satisfactory ceramic to glass seals is that the ceramic have a high thermal shock resistance in order to withstand the heat during the sealing operation. Frequently the ceramic must be gastight, and this may be accomplished by glazing it with a coating of the same glass to which the ceramic is to be sealed.)

See Table I for the linear coefficient of expansion of some refractory materials.

TABLE I. Linear Coefficient of Expansion of Some Refractory Materials.

Material	Temperature range 20° to 400° C.	Temperature range 20° to 600° C.
Silica	0.47×10^{-6}	0.48×10^{-6}
English clay #611	3.7×10^{-6}	4.0×10^{-6}
Zircon	4.3×10^{-6}	4.4×10^{-6}
Mullite	4.8×10^{-6}	6.0×10^{-6}
Alumina	6.8×10^{-6}	7.1×10^{-6}
Beryllia	8.5×10^{-6}	8.4×10^{-6}
Magnesia	11.8×10^{-6}	12.6×10^{-6}
Natural steatite clay	10.8×10^{-6}	10.3×10^{-6}

X

THE COATING OF GLASS AND METAL SURFACES WITH A THIN FILM OF METAL

Glass is coated with a thin reflective film of metal by chemical deposition, burning on, evaporation, and cathode sputtering. Although films of metal may be also produced on metals by these processes, electroplating is the most widely used method for coating metals.

CHEMICAL SILVERING OF GLASS

Choosing Glass To Be Silvered

All glass tubing that is to be used for silvered and evacuated apparatus should be chosen carefully to eliminate tubing with striations. Striations are formed in the drawing of the tube and are air bubbles that have been drawn lengthwise down the tubing. Some of these striations have an opening on the inside of the glass tubing and their capillary action allows acid to be drawn in and expelled during the heating and evacuation, causing the silvered coating to be decomposed. In extreme cases, the striation may open inside the tube at one end and outside the tube at the other end. Such a leak is very hard to locate and the tube is useless for vacuum work.

Checking Glass Tubing for Open Striations

Capillary striations in glass tubing may be tested for leaks or openings inside the tube by means of a colored liquid, such as colored alcohol. The suspected tubing should be filled with the colored solution and allowed to stand for a short period of time. Any striations with openings to the inside will be exposed by the colored liquid drawn up in them because of capillarity.

Cleaning Glass To Be Silvered

One of the most important steps in silvering is the cleaning of the glass. Silver does not deposit on dirty or greasy surfaces, and if the silvered article is to be baked out at high temperatures, it is doubly important that the surface be scrupulously clean. Whenever possible, it is advantageous for the apparatus that is to be silvered to have two tubulations to facilitate introducing the cleaning and silvering solutions. When this is not possible a system such as is shown in Figure 1 may be used. Two separate methods of cleaning may be used, or a combination of both.

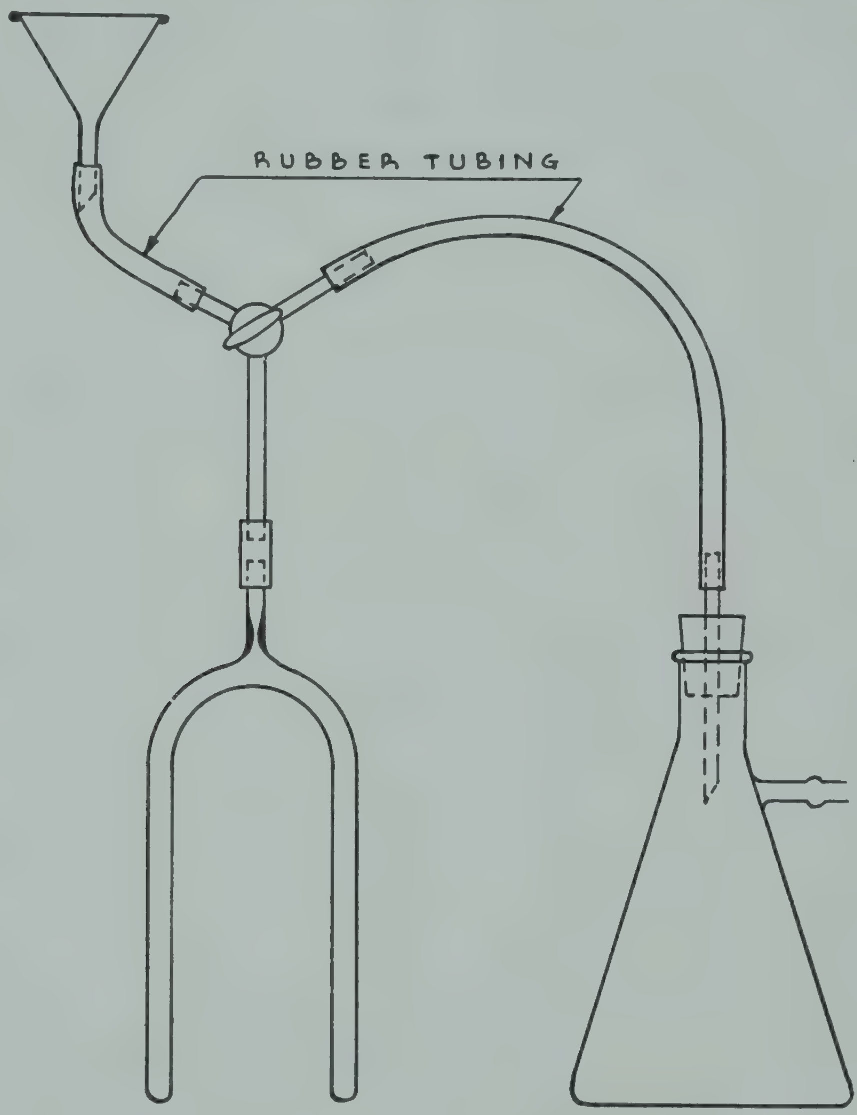

Figure 1. Apparatus for silvering a vessel with one tubulation.

Potassium Dichromate-Sulfuric Acid Solution. The jacket of the vessel is completely filled with the potassium dichromate-sulfuric acid cleaning solution. The temperature of this solution should be about 80° C., and the solution should be maintained at this temperature or at not less than 60° C. for 30 minutes. After this solution is removed and the flask rinsed with distilled water, concentrated nitric acid is introduced into the flask and the flask is shaken for about 10 minutes. After the nitric acid is removed and the flask rinsed at least twice with distilled water, enough concentrated ammonia is introduced to allow the vapor to cover completely the surface of the glass. This action can be clearly observed as the ammonia is added. Enough additional ammonia is then added so that, on shaking, the entire inner surface is washed with the liquid. Without decanting the ammonia from the flask, enough water should be added to fill the flask one-fifth full.After this has been shaken additional water is added until the flask is completely full. This dilute ammonia solution may be left in the flask until the silvering solution is ready, at which time it is drained out and the vessel given a final rinse with distilled water immediately preceding the introduction of the silvering solution.

Hydrofluoric Acid. Scott, Cook, and Brickwedde[1] found that after the standard cleaning procedure of a wash with cleaning solution and nitric acid, a rinse with a 0.7 molal solution of hydrofluoric acid would largely overcome the tendency of the silver to spot out or scale off when the temperature was raised too rapidly during evacuation. The author has found that a 10-minute wash with 0.7 molal solution of hydrofluoric acid is sufficient cleaning by itself for most apparatus requiring silvering. Following the hydrofluoric acid wash, the vessel should be rinsed twice with distilled water and given the same concentrated ammonia treatment as described for the standard cleaning procedure. Contrary to many expressed opinions, a hydrofluoric acid wash will remove ordinary grease and dirt and produce a vessel with a silver coat free from spots and blemishes even after baking.

It is recommended that the hydrofluoric acid wash described above be used only the first time on any vessel. If the silver must be removed and replated a second time, as horter wash (5 minutes) will be sufficient, and if a third plating is required, a wash using standard cleaning solutions of potassium dichromate-sulfuric acid is in order. Too long a wash with hydrofluoric acid or several repeated washes produce a slightly yellow tinge to the silver. If not too pronounced, this will disappear during heating and evacuation; however, a combination of over annealing and excessive washing with hydrofluoric acid may produce a streaked black effect that is permanent.

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[1] Scott, R. B., Cook, J. W., and Brickwedde, F. G., *J. Research, Natl. Bur. Standards*, **7**, 935–943 (1931).

Cleaning Glass with Open Striations. If a completed vessel is found to have striations that open to the inside, complete cleaning may be accomplished by the usual method of acid cleaning and distilled water wash, followed by heating the vessel and boiling concentrated ammonia in it for a short time. This drives the acid from the capillaries and allows them to fill up with ammonia, which will cause no ill effects when liberated during evacuation. For obvious reasons, this boiling should be done in a fume hood if one is available.

Silvering Solution for the Brashear Process

Three solutions are prepared in the following proportions.

A. 2 l. of distilled water, 50 g. silver nitrate.
B. 2 l. of distilled water, 90 g. potassium hydroxide.
C. 800 ml. of distilled water, 80 g. cane sugar plus 100 ml. of ethyl alcohol and 3.5 ml. of nitric acid (specific gravity 1.42).

Solution A should be kept in a dark place or in a dark bottle. Solution C should be aged at least 30 days before using. If it is to be used at once, boiling for 30 minutes will have the same effect as 30 days aging.

The solutions are used in the proportions, A : B : C = 64 % : 32 % : 4 % by volume, or see Table I. The solutions are prepared for use as follows: concentrated ammonium hydroxide is added to solution A, drop by drop, while vigorously shaking the flask until the precipitate, which first forms, completely disappears. To this is added half of the required amount of solution B causing a dark brown or black precipitate to form. If insufficient ammonium hydroxide is added before solution B is added, the precipitate will be a yellowish green. If this does occur, the solution should be discarded and the process started again. Ammonium hydroxide is added again, drop by drop, while shaking

TABLE I. Silvering Solutions

Amount of solution needed, ml.	Solution A, ml.	Solution B, ml.	Solution C, ml.
50	32	16	2
100	64	32	4
150	96	48	6
200	128	64	8
250	160	80	10
300	192	96	12
350	224	112	14
400	256	128	16
450	388	144	18
500	320	160	20
550	352	176	22
600	384	192	24
650	416	208	26
700	448	224	28
750	480	240	30
800	512	256	32
850	544	272	34
900	576	288	36
950	608	304	38
1000	640	320	40
1100	704	352	44
1200	768	384	48
1300	832	416	52
1400	896	448	56
1500	960	480	60
1600	1024	512	64
1700	1088	544	68
1800	1152	576	72
1900	1216	608	76
2000	1280	640	80
2200	1408	704	88
2400	1536	768	96
2600	1664	832	104
2800	1792	896	112
3000	1920	960	120
3500	2240	1120	140
4000	2560	1280	160
4500	2880	1440	180
5000	3200	1600	200

until the black precipitate is almost, but not completely, dissolved. Gradually the remaining half of solution B is added, while continuing to clarify the solution with ammonium hydroxide. The ammonium hydroxide should be added more slowly toward the last, since the reaction is slow and there is danger of going past the end point. The final solution should not be completely clear; rather it should be a slightly cloudy solution which if allowed to stand for 30 minutes, will become clear except for a few black flakes which settle to the bottom. The solution may be used immediately or it may be allowed to stand for as long as an hour without affecting the results.

Cooling Silvering Solutions. After solutions A and B have been prepared by the addition of ammonia hydroxide, it is advantageous to cool them to at least 15° C. For strip silvering, a temperature of 10° to 12° C. is recommended. The lower temperature allows time for decanting the solution into the vessel after the addition of solution C and before the actual plating out begins.

Silvering Procedure

When no window is required, enough solution should be prepared to fill the vessel about two-thirds full. This solution may be decanted into the vessel, the required amount of solution C added, and the solution mixed by shaking. The vessel should be rolled or shaken during the entire plating operation. The time required for the solution to react and deposit the silver on the walls depends on two factors: (1) the temperature of the solution (the lower the temperature, the slower the reaction); (2) the age of solution A(the older the solution, the slower the reaction). The time of completion of the silvering process can be determined by occasionally removing a little of the solution and noticing the character of the precipitate. When the solution becomes flocculent the silvering is completed and the solution should be removed to prevent the formation of a gray deposit or bloom. With practice, the condition of the solution inside the vessel can be determined by feel. When the solution is spent, the precipitate forms a heavy sludge that can actually be felt moving from one end of the vessel to the other as it is tilted end to end.

If a heavy coat of silver is desired, a second coat may be deposited over the first after removing the spent solution. Brighter and thicker silver coatings may be obtained with a given amount of solution by silvering twice with half-strength solutions (obtained by diluting with distilled water) than can be obtained by silvering once with the full-strength solution.

After silvering is complete, the vessel is washed out and as much as possible of the sediment adhering to the walls is removed by a vigorous shaking with the vessel half full of water. After a final rinse with distilled water and a rinse with methyl alcohol, the vessel is dried by alternately exhausting and filling with dry air. The pump used in this operation should be carefully trapped to avoid contaminating its oil with alcohol.

Partial Removal of Silver

The partial removal of silver without affecting the rest of the silver may be accomplished with the following solutions:

A. 5 % solution of cupric chloride.
B. 15 % solution of sodium hyposulfite.
C. 10 % solution of sodium hydroxide.

Solution A is decanted into the vessel to the predetermined line to which the silver is to be removed. If it is difficult to look inside the vessel and see the amount of solution added, solution A can be let in slowly until the gradual decomposition of the silver has been observed to reach the proper line. After the silver has become discolored brown in appearance, Solution A may be removed, the vessel washed with distilled water twice, and solution B added, taking care to bring the level of solution B even with that of solution A. When the silver has been completely removed, solution B is removed and the vessel is rinsed with distilled water. If some particles of silver remain on the glass, the above process is repeated with both solutions A and B. When the removal of silver is complete, solution C is used for a neutralizer, with two rinses with water. The apparatus is then ready for use.

Strip Silvering

On occasion it is desirable to plate only part of a jacket surface or, in the case of distillation equipment, to leave a window of the unsilvered glass running the length of the column. To do this, the jacket is supported on two stands and adjusted to a level position with the aid of a spirit level. Two tubulations are required and the solution allowed to flow by gravity into the jacket until it reaches the required level. See Figure 2. To avoid uneven plating, solutions A and B are prepared and cooled in an ice bath to 10° to 12° C. At this temperature the plating action started by adding solution C is delayed enough for the combined solution to flow into the jacket. Having the solution cooled also increases the sharpness of the line or edge of the window, which is desirable. After the silvering is completed on one side, the column is thoroughly washed with water and inverted and the silvering process repeated (see Figure 2B).

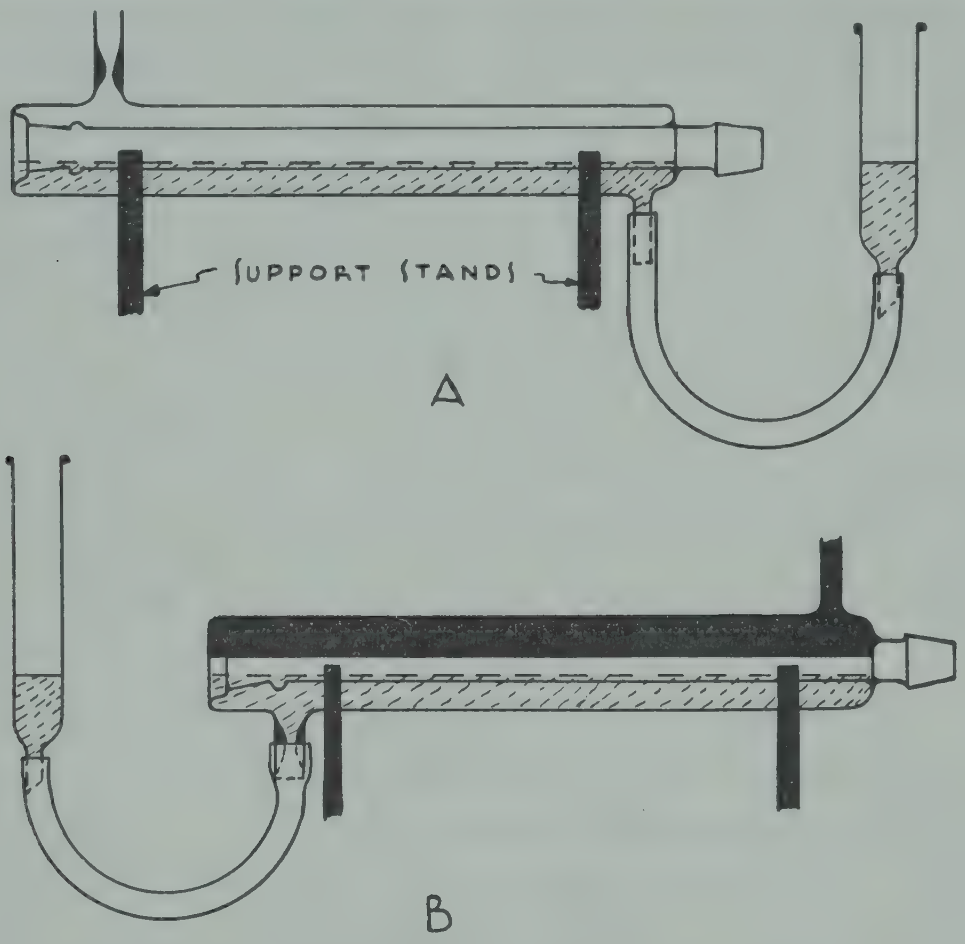

Figure 2. Procedure for strip silvering.

When the solutions are not cooled, it is necessary to dry the inside of the jacket completely before the silvering solution flows into the jacket in order to insure a straight even edge to the window. (Uncooled solutions have a tendency to creep up a wet surface of glass, resulting in a streaked uneven line.)

Cleaning Glass Preparatory to Working

Glass that is ultimately to be silvered should be given a scrupulously careful cleaning. Finger marks, grease, and dirt burned into the glass during the glassblowing operations are more clearly shown than in unsilvered glass.

Cleaning Glass for Mirrors

Glass to be silvered for use as a mirror requires nearly as thorough a cleaning as that for evacuated vessels. If the mirror blank is small enough to put in a beaker or tray, the same chemical cleaning solutions used for vacuum jacketed vessels may be used. On large plates of glass the cleaning procedure described in steps A to C below may be used satisfactorily.

A. The grease is removed by swabbing the entire surface with a cotton swab soaked in alcohol.

B. The entire surface is covered with nitric acid and allowed to stand for 15 to 20 minutes, taking care to pour onto the surface only the amount of acid that can be held on the surface of the glass without running over the edges.

C. The acid is removed and the surface of the glass rinsed off with distilled water. The entire surface is then carefully swabbed with a piece of damp cotton and powdered calcium carbonate.

Step A may be replaced by a wash with Aerosol (made by Proctor and Gamble and described by Duncan[2]), a detergent made of sulfonated organic compounds of high molecular weight. It has a neutral reaction and may be used in either neutral, caustic, or acid solutions. Unlike soap, it forms a soluble compound with magnesium and calcium compounds which are common in tap water. The detergent Dreft, obtainable at grocery stores, is also satisfactory for washing glass for mirrors. The glass should never be allowed to dry during the cleaning operation and should be covered with distilled water until actual plating begins.

Silvering Small Mirrors by the Brashear Process

Solutions A and B are prepared as previously described. The reducing agent (C) is added and the silvering solution poured over the mirror blank. When the solution develops a muddy brown appearance, deposit of silver on the mirror has reached a continuous stage. The container should be tipped from time to time to stir the solution and allow for inspection of the surface. When the silver film covers the whole surface, and black specks begin to settle on it, a light swabbing with a cotton pad is recommended. The swabbing is done lightly at first, but may be more vigorous as the silver becomes thicker, and the surface is checked from time to time for the appearance of bloom. When the solution begins to clear up, the solution is nearly spent and should be discarded, and the mirror rinsed with distilled water.

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[2] Duncan, R. A., *Ind. Eng. Chem.*, **26**, 24 (1934).

When a satisfactory coat has been obtained, and after it is dry, it may be burnished and finally polished with a chamois skin and optical rouge. The rouge pad may be used from time to time to polish away tarnish which forms on the silvered mirror.

Silvering Large Mirrors

After cleaning, large mirrors may be silvered by pouring silvering solution (A, B, and C) onto the surface of the glass, taking care to pour only as much of the newly mixed solution as can be held on the surface of the glass without running over the edges of the glass. When the solution begins to clear up, the glass is raised and the liquid allowed to drain off. This is followed by washing the surface with distilled water. Mirrors made this way are likely to have small pinholes in the silver resulting from particles of sediment depositing from the solution. After rinsing the surface with distilled water, a second coating of silver will cover the imperfections left in the first coat.

Rochelle Salt Process

Two solutions are required for the Rochelle salt process. Solution A is made as follows. Five grams of silver nitrate is dissolved in 500 ml. of water and titrated with ammonia until a distinct straw color remains. If it accidentally becomes clear, it should be back-titrated with a dilute solution of silver nitrate until the straw color reappears.

Solution B consists of 5 g. of silver nitrate dissolved in

500 ml. of water. It is brought to a boil and 0.83 g. of Rochelle salt, dissolved in a little water, is added. Boiling is continued until a gray precipitate is deposited. The solution is filtered while it is still hot and diluted to 500 ml.

To silver a mirror, solutions A and B are mixed in equal volumes and poured directly onto the mirror blank. The temperature recommended for silvering is 20° C.

Silver is deposited slowly by the Rochelle salt process, with 1 hour being required for a thick coat to deposit. Partial reflecting mirrors are obtained by removing the glass from the solution when the required degree of reflectivity has been obtained. The progress of plating may be followed by removing small sample plates from time to time and inspecting them.

Solutions A and B, as described above, may be kept stored for as long as one month if protected from the light.

Lacquering Silver Surfaces

Silver mirrors may be protected from tarnish by covering them with a thin layer of colorless lacquer. The lacquer tends to destroy some of the reflectivity of the silver and, in addition, shows some interference colors, although R. Wood[3] states that a thin transparent film of lacquer on a good mirror surface should not show interference colors. The colors usually observed are due to frilling, which can only be observed directly under a high-power microscope. To avoid frilling, Wood recommends a coating of collodion dissolved in chemically pure redistilled ether. To obtain uniform lacquer films with collodion and ether, it is necessary that the ether evaporate slowly.

Painting Glass Mirrors

After the silvered surface is dry, it should be given a protective coating of asphaltum varnish. This will make the surface less likely to be affected by atmospheric changes and also acts to prevent scratches and other injuries from handling. The brush with which this is applied should be free from grit and soft, so that there is no danger of scratching the surface during the painting.

[3] Wood, R. W., *Physical Optics*, 3rd ed. Macmillan, New York, 1934.

PLATINIZING GLASS

Glass may be coated with a thin layer of platinum by the burning-on method. The glass is coated with a layer of oily solution of platinum salt. When heat is applied the oil burns away, leaving a deposit of metal. This deposit is firmly bonded to the glass by a final heating close to the softening point of glass. After this the deposit may be electroplated with copper and soldered to metal to form a vacuum-type seal.

A platinum film may also be burned onto porcelain. After copper plating this may be soldered to metal, thus making a vacuum-tight seal between porcelain and metal.

Platinizing Solution

A solution prepared as follows will give satisfactory results.

A. One part of pure platinic chloride, prepared by evaporating a 10 % solution to dryness, is moistened with a few drops of absolute alcohol and ground in an iced mortar with 10 parts lavender oil. The lavender oil is added gradually in small quantities. Burgundy pitch is then added to the solution until the latter has the desired consistency. Ordinary rosin may be substituted for the Burgundy pitch with no deleterious effects.

B. A second platinizing solution is as follows:

	Parts by volume
Collodion in methyl alcohol, 6 % solution	3
Chloroplatinic acid in denatured alcohol, 6 % solution	3
Denatured alcohol	3
Bismuth chloride	1

The bismuth chloride is made from a 5 % solution of bismuth chloride in denatured alcohol to which 5 % of hydrochloric acid is added. This solution is stored and diluted to the proper proportion just before use.

When heated, the collodion chars and completely volatilizes, leaving a film of platinum. Continued heating produces a platinum film which adheres to the glass but is soft. Eventually after being heated again the platinum film becomes incorporated with the surface of the glass and is permanent. In fact it will be necessary actually to scratch the glass to make a scratch mark in the platinum.

Notes. For coating glass as a preliminary step in soldering to metal, the consistency of the platinizing solution is of little importance. Platinizing solutions which have been kept for a long period of time gradually thicken when exposed to air; however, since no platinum has been deposited, the solution can be used to produce satisfactory results.

CHEMICAL PLATING OF GOLD

In 1868 Wernicke[4] developed a method of making mirrors by plating gold on glass. This had become somewhat forgotten by 1926, when Von Angerer[5] republished the formulas in detail.

Gold Plating Solutions and Procedures

Three solutions are necessary for goldplating and these may be made up and stored for a long period of time.

Solution No. 1 is a solution of gold chloride ($AuCl_3$) of such concentration that each 120 ml. of water contains 1 g. of pure gold. It is important that the solution be free from any other metal such as silver, which would plate out along with the gold when the reducing agent is added.

Solution No. 2 is a sodium hydroxide solution having a specific gravity of 1.06. This need not be pure.

Solution No. 3 is the reducing agent, and is prepared by mixing 50 g. concentrated sulfuric acid, 40 g. of alcohol, 55 g. of distilled water, and 50 g. of powdered manganese dioxide. This mixture is gently heated in a water bath and the vapor evolved is passed through 50 ml. of cold water. The gentle heating is continued until the volume of water has doubled (i.e., until 100 ml. are obtained). To this 100 ml. of acetaldehyde solution (CH_3CHO) 100 ml. of alcohol and 5.0 g of glucose are added. The solution is then made up to 500 ml. by adding distilled water.

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[4] Wernicke, *W.*, *Pogg Ann.*, **133**, 183 (1868).

[5] Von Angerer, Ernst, *Wien–Harms Handb. exptl. Physik*, **1**, 375 (1926).

When cane sugar is used in place of glucose, it should be prepared by dissolving 10 g. of sugar in 70 ml. of water, adding 0.5 g. of $4N$ nitric acid, and then boiling the solution for 15 minutes. The resulting solution is added to the 100 ml. of acetaldehyde solution, plus the 100 ml. of alcohol, and diluted to 500 ml.

To gold plate a piece of glass a portion of the sodium hydroxide solution is mixed with four times its volume of gold solution (No. 1), to which is added $^1/_{35}$, or at the most $^1/_{30}$, by volume, of the reducing solution.

After the reducing agent has been added, the solution quickly turns green owing to the formation of colloidal metallic gold. This solution should immediately be brought into contact with the surface to be plated. At ordinary room temperatures the deposit begins to form in about 30 minutes. After 1½ hours it is transparent, with a magnificent green color. After 2½ to 3 hours the plating should be thick enough to be scarcely transparent.

Warming the solution will increase the speed of plating; however, a temperature above 75° C. is not satisfactory because the gold plating seems to adhere less firmly to the glass. Within the stated limits, the quality of the gold plating, in respect to its smoothness and stability, is the same whether it is plated at room temperature or with a warmed solution. However, if the solution is warmed, it is advisable to heat the alkaline gold solution to almost boiling to remove air which otherwise would rise in small bubbles and produce small holes in the plating. These holes are not evident in the reflection but are shown by transparency. The gold plating obtained is thoroughly washed with water, allowed to drain on filter paper, and dried at ordinary room temperatures.

Cleaning the Glass Surface To Be Gold-Plated

The preparation and cleaning of the surface of the glass to be plated are the same as for silver plating. Before the actual plating operation, however, the surface of the glass should be rinsed with an alkali followed by distilled water.

CHEMICAL PLATING OF COPPER

The deposition of a brightly reflective copper mirror from a solution of its salts is considerably more difficult than silver plating.

Copper films were first obtained by Faraday in 1857 when he produced a mirror by heating a plate of glass in a mixture of copper oxide and olive oil. Unfortunately mirrors obtained by this method are both uneven in thickness and poor in reflectivity.

For the successful chemical plating of copper mirrors French[6] found that complete accuracy in the proportions used for preparing the solutions and precise control of the temperatures at which the plating is done are very important.

Copper Plating Solutions and Procedures

The two solutions required are mixed as follows:

Solution 1. Nine grams of hydrazine sulfate is dissolved in 150 ml. of distilled water and heated to 60° C. With constant stirring, 90 ml. of a saturated solution of copper hydroxide and ammonia (at 16° C) is added. The resulting mixture will be a clear yellow color. The glass to be plated should be sponged with this solution after undergoing the cleaning process outlined later. This sponging is very important and was found to be necessary to obtain satisfactory mirrors.

Solution 2 consists of 11.1 g. of potassium hydroxide (purified by alcohol) to each 100 ml. of distilled water. This solution is heated to 60° C., and 87.5 ml. of this hot solution is quickly added to solution No. 1 while stirring constantly. If these procedures are carried out correctly the resulting solution should still be a clear yellow color with no precipitate.

This mixture of solutions 1 and 2 is then poured into a copper vat (Figure 3), which is heated to 43° C, in a water bath. The glass blank to be plated should be immediately suspended in the solution and periodically given a rotary motion in the liquid. The temperature of the water bath should be gradually raised to 57° C. and held there. When solutions 1 and 2 are mixed, the resulting solution will turn a dark green and remain that color for about 15 minutes, after which the color will commence to turn pink. The complete change should take about 20 minutes, by which time the copper film will have formed on the surface of the glass.

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[6] French, E., *Trans. Opt. Soc. (London)*, **25**, 229 (1924).

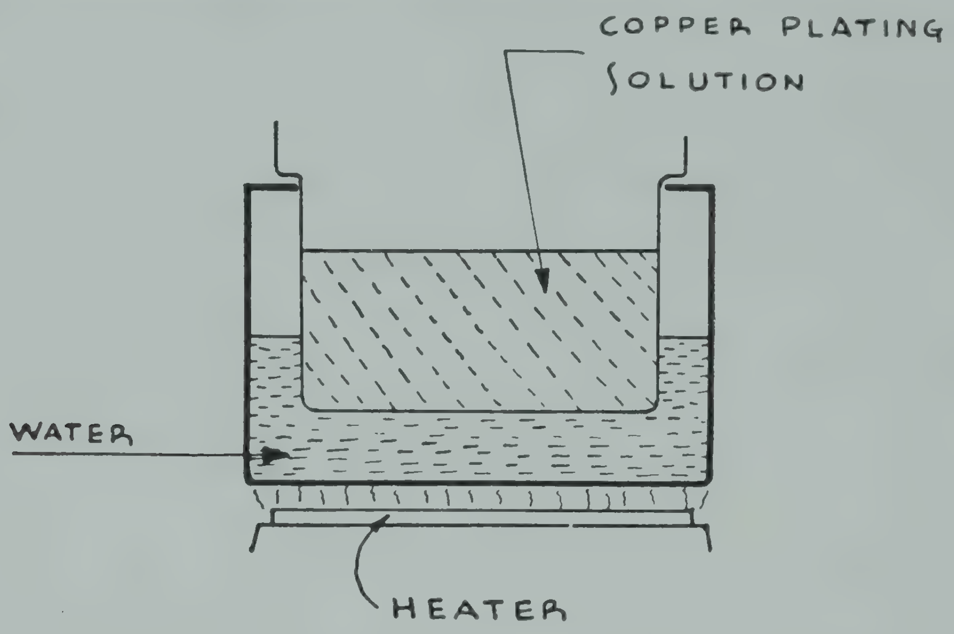

Figure 3. Vat for copper plating on glass.

The copper film at this stage is very thin, easily scratched, and quick to oxidize. To protect it the following method of thickening the copper plate is used. The mirror is left in the first plating solution an additional 5 minutes, after which the temperature of the plating solution is lowered to 43° C. To this cooled solution, a refresher solution is added. The refresher consists of 50 ml. of a solution of copper hydroxide and ammonia (at 16° C.) to which 10 ml. of a solution of potassium hydroxide (at 60° C.) has been added. The refresher mixture is added to the partially spent copper-plating solution by quickly lifting the mirror from the solution and pouring in the refresher while constantly stirring. When the new solution has been added and thoroughly mixed, the mirror is again lowered into the plating solution. The temperature of the revived solution should be about 43° C. After the glass mirror has been immersed in the plating solution, the solution turns black again. Further reduction of copper will take place if the temperature is gradually raised to 50° C., thus thickening the initial plate.

By exactly the same process used previously, the temperature is again lowered to 43° C. and a second refresher solution, consisting of 1 g. of hydrazine sulfate dissolved in 60 ml. of a solution of copper hydroxide and ammonia (16° C.), is added. When this addition is complete, the temperature is raised to 50° C. during which time the color of the resulting mixture changes. After 15 minutes the temperature is again lowered to 43° C. and another portion of refresher 1 is added (i.e., 50 ml. of copper hydroxide and ammonia solution at 16° C., to which 10 ml. of potassium hydroxide at 60° C. has been added.). After a period of 15 minutes, during which time the temperature is raised to 50° C, the solution is again cooled to 43° C. and a second portion of refresher 2 is added (1 g. of hydrazine sulfate dissolved in 60 ml. of the copper hydroxide-in-ammonia solution at 16° C). After the temperature of the vat is again raised to 50° C., the whole is allowed to cool to room temperature before the mirror is finally taken out.

Thickening Copper Film by Electroplating

As a final protection the copper mirror, after being washed in ordinary and distilled water, is dried and placed in an electroplating bath and copper is electrically deposited for ½ hour. *Caution:* this electroplating must *not* be done in an ordinary cyanide or acid copper sulfate bath, since such baths cause the copper film to strip off. The following bath may be used with satisfactory results.

One part by weight of potassium bitartrate is boiled in 10 parts (by weight) of water. As much hydrated carbonate of copper as will dissolve in the liquid is then added. The final mixture is then made alkaline by the addition of a small quantity of potassium carbonate.

A safe current to use is 0.25 amp. per square decimeter. If the current density at the cathode becomes too great, there is a danger of stripping the film.

The whole deposit may be protected by a lead backing if desired.

Cleaning the Glass Surface To Be Copper-Plated

The glass surface is thoroughly cleaned with concentrated nitric acid, and then after being washed with tap water it is placed in a warm solution of 1 % caustic potash for ½ hour. It is then taken out and washed for about half an hour in tap water, and given a final wash with distilled water. The whole surface is then thoroughly polished with a perfectly clean, dry cloth, which has been previously washed in the following manner.

The cloth is first boiled in water with Castile soap, after which it is removed with metal tongs and boiled in successive changes of water until no trace of milkiness is perceptible. This method is laborious, but it is very efficient for removing grease. The wash cloth after drying should be protected from grease, handled with tongs alone, and used only once before rewashing.

PLATING BY EVAPORATION

This method involves the evaporation of metal in such a degree of vacuum that the mean free path of the molecules is longer than the diameter of the evacuated chamber. In practice a small strip of the metal to be evaporated is placed on a tungsten

TABLE II. Evaporation Temperatures of Metals

Material	Evaporation temperature, T, °Absolute *	Material	Evaporation temperature, T, °Absolute *
Hg	320	Pb	1000
Cs	433	Sn	1148
Rb	450	Cr	1190
K	480	Ag	1319
Cd	541	Au	1445
Na	565	Al	1461
Zn	623	Cu	1542
Mg	712	Fe	1694
Sr	811	Ni	1717
Li	821	Pt	2332
Ca	878	Mo	2755
Ba	905	Co	2795
Bi	913	W	3505
Sb	973		

*Temperature at which the vapor pressure equals 10^{-2} mm. of mercury.

heater and brought to the evaporation temperature as shown in Table II. For most purposes the pressure should be between 10^{-2} and 10^{-4} mm. of mercury.

Evaporation Chamber and Base Plate

The evaporation chamber is usually a heavy glass bell jar, resting on a metal base plate. As a safety feature, the bell jar should be surrounded with a wire screen for protection in case of an implosion during the evacuation. The metal base plate should have a large central hole through which the bell jar is evacuated, and there should be provision for at least two insulated electrodes to carry the current for heating the evaporation sources. A commercial base plate, which may be obtained from Central Scientific Co.,[7] has four insulated lead-ins provided for attaching evaporation sources, and a fifth highly insulated lead-in for the high-voltage discharge which may be used for the final cleaning of the surfaces to be plated. The discharge induced by this fifth lead-in may also be used as an indication of the degree of vacuum.

Evaporation Source

The type of evaporation source used depends on the metal being evaporated. The metals most widely used for evaporation sources are tungsten, molybdenum, or tantalum.

Tungsten Helical Coils. Strong[8] found that tungsten has a limited solubility for molten aluminum, and because of this the most practical method of evaporating aluminum is from tungsten coils (Figure 4). The aluminum, which melts before it evaporates, is kept from falling out of the coil by surface tension. Some metals such as magnesium sublime. Chromium, which sublimes very slowly, must be prefused to the tungsten wire by being heated to fusion temperature in an atmosphere of hydrogen or helium. These gases increase the heat transfer between the chromium and the tungsten and also reduce the evaporation of the chromium. Chromium may also be electroplated on the tungsten coil and then evaporated. This method of electroplating the coils also works well with platinum and beryllium.

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[7] Central Scientific Co., 1700 Irving Park Road, Chicago 13, Illinois.
[8] Strong, J., *Phys. Rev.*, **43**, 498 (1933).

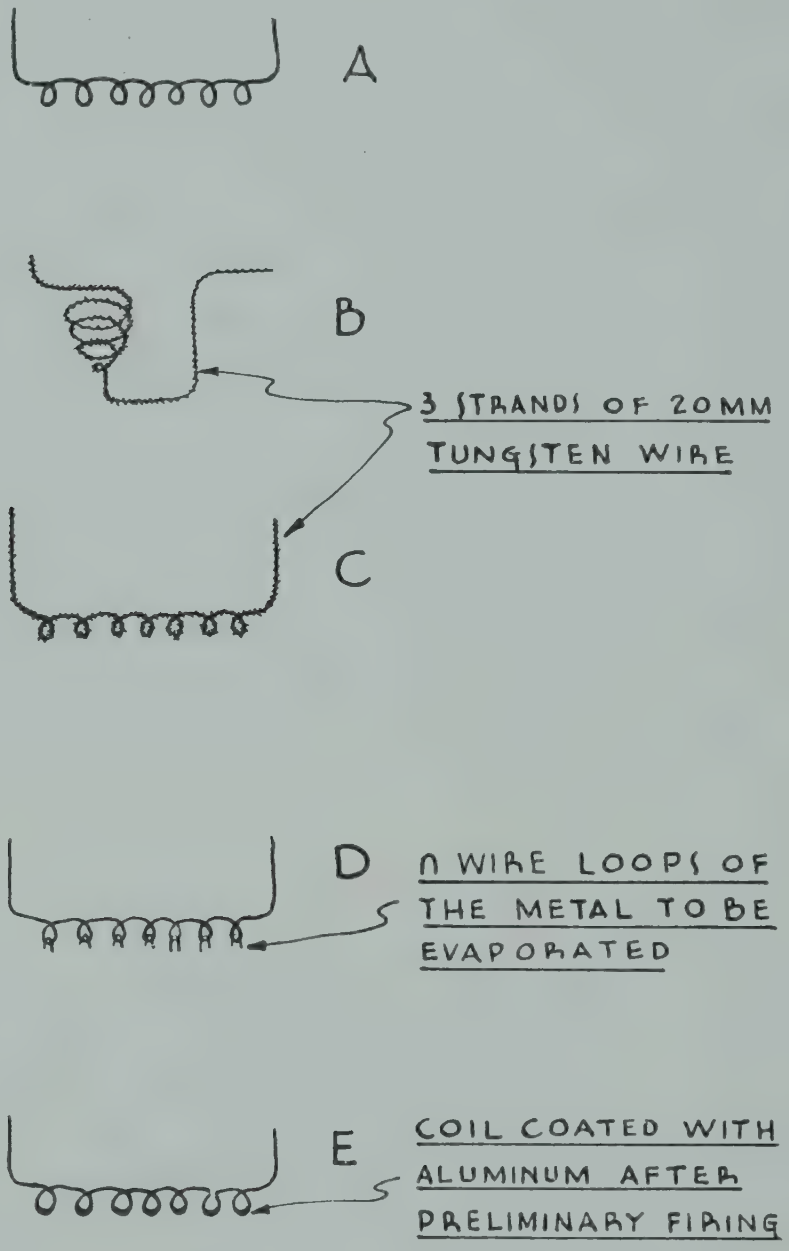
A
B
3 STRANDS OF 20MM TUNGSTEN WIRE
C
D
n WIRE LOOPS OF THE METAL TO BE EVAPORATED
E
COIL COATED WITH ALUMINUM AFTER PRELIMINARY FIRING

Figure 4. Tungsten helical evaporators.

Strip or Boat Heater. An evaporation boat is shown in Figure 5A and B. If these boats are made of molybdenum or tantalum they may be stamped cold. Tungsten boats must be stamped and cut with the metal heated to about 400° C.

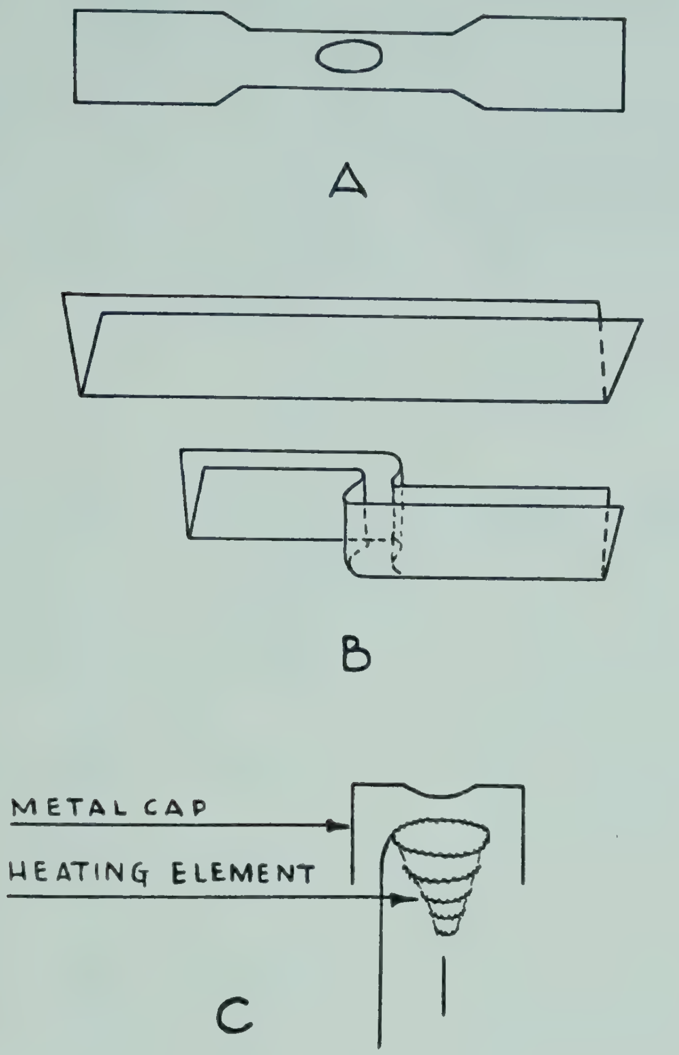

Figure 5. Evaporating boats.

Evaporation Cap. Figure 5C shows a small cap which is placed over a heater wire. The metals to be evaporated are placed in the small cup in the top.

Evaporation Procedure

The evaporation of most materials may be carried out with a vacuum system containing an oil diffusion pump, without trapping. When a mercury diffusion pump is used, a liquid air trap is recommended during the evaporation of aluminum. For other metals a carbon-dioxide–alcohol trap is sufficient. Table III shows the evaporation source best suited for a number of metals, and Figure 6 shows an apparatus for plating by evaporation.

TABLE III. Evaporation Sources for Metals

Metal	Source	Procedure
Aluminum Magnesium Cadmium Beryllium Calcium	Tungsten helix (Figure 4A)	These metals should be prefused to a tungsten helix under an atmosphere of hydrogen or helium
Aluminum Nickel Iron Cobalt	Tungsten helix (Figure 4A)	A strip of the metal to be evaporated may be crimped to the tungsten helix and prefused to the helix, or it may be electroplated to the helix
Chromium Gold	Tungsten helix (Figure 4B)	A piece of chromium or gold is placed in the cup and prefused in an atmosphere of hydrogen or helium
Oxides	Tantalum cap (Figure 5C)	Material to be evaporated is placed in the dent in the cap and heated
Platinum	Tungsten helix (Figure 4C)	Helix is made of three strands of 20-ml. tungsten wire and one strand of 20-ml. platinum wire wound together Platinum may also be electroplated to a solid tungsten wire helix
Rhodium	Tungsten helix (Figure 4A)	Rhodium should be electroplated to the tungsten helix. Liquid air trapping is advisable
Silver Copper	Molybdenum or tantalum boat or strip Figure 5A and B	Tungsten is not satisfactory for the source since it is not easily wet by either copper or silver
Salts Silica Nonmetals	Metal boat or strip or tungsten helix	If a tungsten helix is used, the material being evaporated should be electroplated to the helix

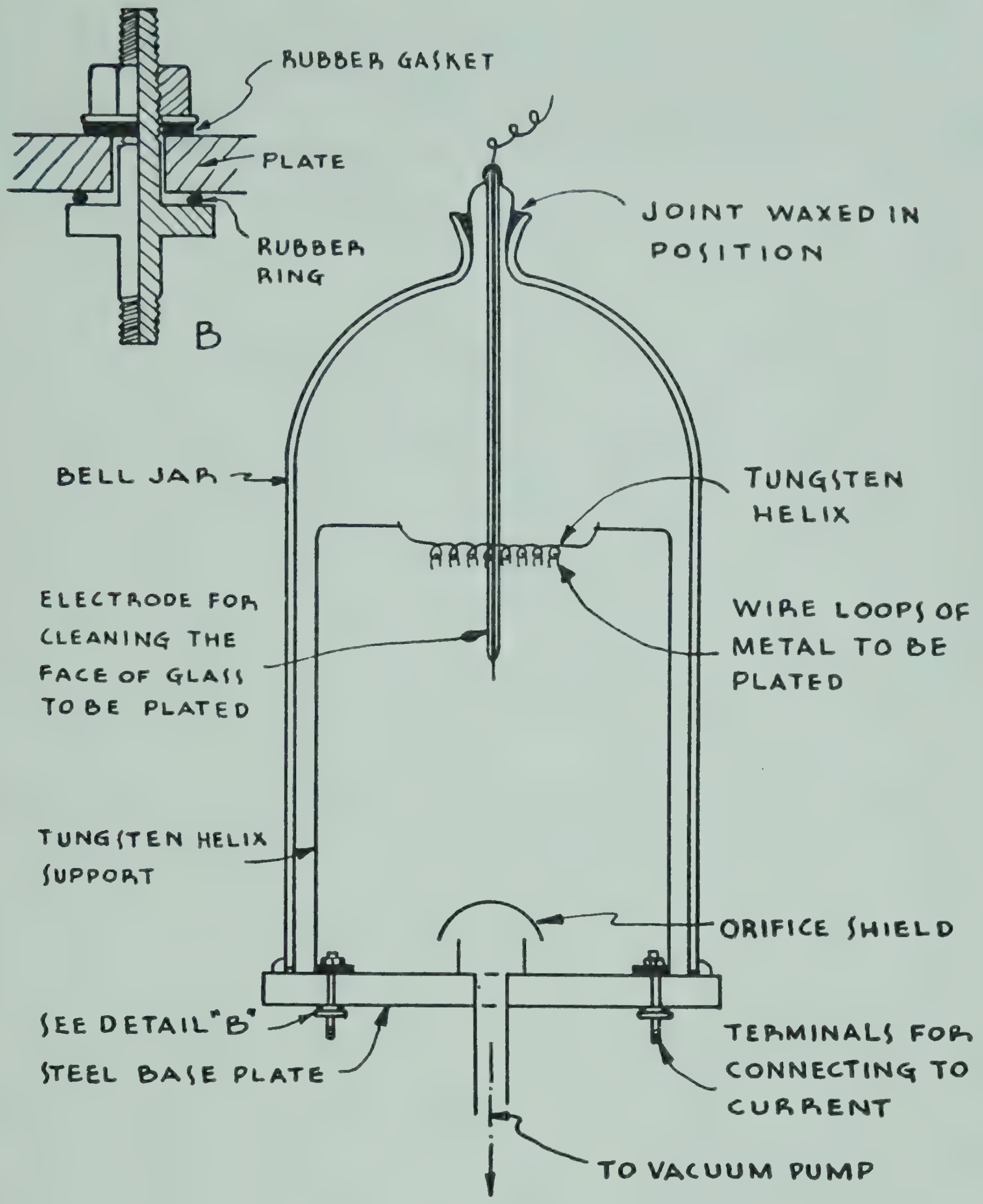
RUBBER GASKET
PLATE
RUBBER RING
B
JOINT WAXED IN POSITION
BELL JAR
TUNGSTEN HELIX
ELECTRODE FOR CLEANING THE FACE OF GLASS TO BE PLATED
WIRE LOOPS OF METAL TO BE PLATED
TUNGSTEN HELIX SUPPORT
ORIFICE SHIELD
SEE DETAIL "B"
STEEL BASE PLATE
TERMINALS FOR CONNECTING TO CURRENT
TO VACUUM PUMP

Figure 6. Apparatus for plating by evacuation.

Cleaning Surface To Be Plated

The proper cleaning and drying of surfaces to be plated by evaporation is more critical than the cleaning of surfaces to be chemically plated. The procedure is the same as that outlined for cleaning surfaces for the chemical plating of silver. Much of the difficulty in preparing surfaces for plating by evaporation arises in the drying of the surface after it has been cleaned. Satisfactory results are obtained by drying the surface with an absorbent cotton towel which has been well boiled and soaked in benzene to remove any possible grease. Surfaces may also be dried in a desiccator after the chemical cleaning. The final step in cleaning a surface is to subject it to a glow discharge in the vacuum system. Aitken[9] and Rayleigh[10] found that the surface of glass can also be cleaned by passing a hot flame quickly over it. According to Strong,[11] the ions from the flame and glow discharge act to clean the surface of the glass.

PLATING BY SPUTTERING

In sputtering, particles of metal from the cathode are thrown out impinging on a surface placed in their path. The cathode is made of the metal to be sputtered and the anode is usually made of aluminum or iron. The alternating current from a 10,000-volt neon-sign transformer may be used as the source for the sputtering potential, although other sources such as batteries or induction coils may be used.

The phenomenon of sputtering was discovered in 1852 by Grove.[12] It is still not completely understood and there are two current theories as to its cause. One theory states, "the emission of metal by the cathode is pure thermal evaporation, due to the high temperature attained in areas of molecular dimensions. These high temperatures are produced by the energy of the impinging ions." The second theory suggests "a mechanism for transferring the energy of the gas ion into energy of a metal molecule which is similar to the mechanism by which the energy of light is transferred to energy of an emitted electron."

Sputtering Procedure

Plating by the sputtering method may be carried out at pressures ranging from 1 to 10^{-2} mm. of mercury. The pressure usually chosen is that which gives a dark space about the same length as the distance from the cathode to the surface to be plated.

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[9] Aitken, *Royal Soc. Edinburgh*, 94, (1893).

[10] Rayleigh, J. W. S., *Scientific Papers*, **6**, 26 (1920).

[11] Strong, J., *Procedures in Experimental Physics*, p. 167. Prentice-Hall, New York, 1939.

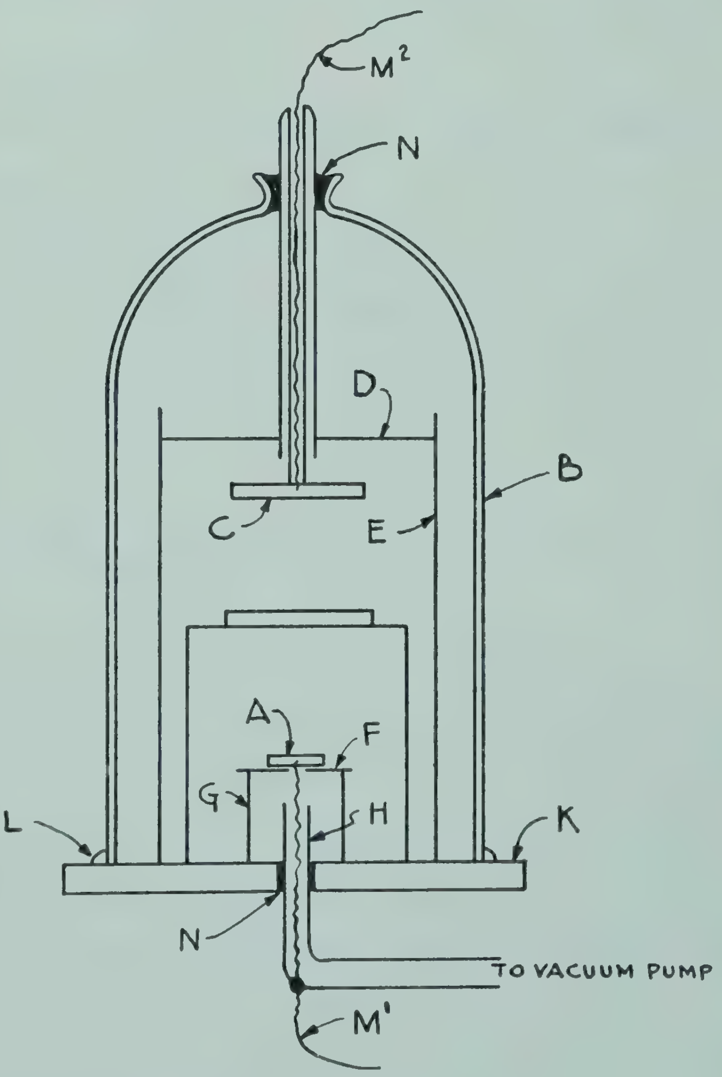
[12] Grove, W. R., *Phil. Trans.*, **1**, (1852). Taken from Strong, J., *Procedures in Experimental Physics*, p. 159. Prentice-Hall, New York, 1939.

The residual gas in the bell jar may be air, nitrogen, argon, or hydrogen. Helium produces a very low sputtering rate and is generally only used for glow discharges when sputtering is not desired.

Notes on Cathode

The size and shape of the cathode depends on the surfaces to be plated. A U-shaped cathode may be used to plate both sides of a mirror at once. A cylindrical cathode may be used for coating the outside of a tube or a wire, while a central wire cathode may be used to plate the inside of a tube, providing the length of the tube does not exceed its diameter. For plating curved mirrors, the cathode should be shaped to follow the same curvature as the mirror.

In 1934 Hulburt[13] made a study of sputtering. He used a voltage between 1000 and 3000 volts and a current of 50 milliamperes, and the sputtering was carried out at a pressure which



A — Anode
B — Bell Jar
C — Cathode
D, E — Glass shields to protect bell jar from being plated
F — Glass support for anode
G — Glass support for F
H — Glass tube to vacuum pump
K — Metal base plate
L — Apiezon Q, vacuum seal
M^1, M^2 — Sealed-in electrodes
N — Vacuum wax

Figure 7. Apparatus for sputtering.

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[13] Hulburt, E. O., *Rev. Sci. Instr.*, **5**, 85 (1934).

resulted in a 5 cm. dark space in a residual atmosphere of air. Table IV gives his results, while Figure 7 shows an apparatus for sputtering.

TABLE IV. Time To Obtain Films by Sputtering

Time to produce an opaque coating, hours	Metals to be sputtered
1	Antimony, bismuth, cadmium, gold, lead, platinum, silver, tin, zinc
2	Cobalt, copper, iridium, iron, nickel, selenium, tellurium
Several	Molybdenum, tantalum, tungsten
15[1]	Aluminum, beryllium, carbon, chromium, magnesium, silicon

[1] To speed the sputtering time of these metals to 15 hours it is necessary to introduce mercury vapor, helium, or argon to the sputtering chamber.

Cleaning Glass Surfaces for Sputtering

The same procedure for cleaning glass surfaces preparatory to plating by evaporation should be followed to clean a surface for sputtering. Care must again be exercised during the drying. Final cleaning is accomplished by means of a glow discharge in a vacuum.

ELECTROPLATING

The process of depositing a coating of metal upon any object by means of an electrical current is termed electroplating. The metal held in solution is deposited on the object to be plated which is connected to the negative pole of the source of electricity. A plate of metal from which the coating is being made is used as the positive plate. Electroplating is based on the principles of electrolysis, as expressed in Faraday's laws, which are as follows:

A. Chemical changes during electrolysis occur only at the surface of the electrodes.

B. The amount of chemical decomposition, measured by the weight of substances liberated during electrolysis, is proportional to the current and the time for which it flows, i.e., to the quantity of electricity which passes through the solution.

C. If the same quantity of electricity is passed through a number of electrolytes, the weight of the substances liberated is proportional to their chemical equivalents.

Preparation of Metal Surfaces for Electroplating

In electroplating it is almost impossible to obtain a satisfactory plating if the surface has scratches, pits, or cuts filled with grease or metallic filings. It is also very important that the surface be free from rust, since rust causes areas in which peeling will start. Cleaning is accomplished by grinding, polishing, and chemical cleaning.

Grinding. Grinding is the term used for the operation of removing coarse irregularities from the surface of the metal, prior to the final polishing, and this is usually done with an emery wheel.

Polishing. Polishing is the finishing process used prior to the metal finishing operation. With the modern methods of polishing, emery is used for the fast-cutting abrasive and tripoli, rouge, and lime are used for the smoother cutting. Polishing wheels are often made from soft felt, blended to give uniform texture and resiliency. Such wheels are called polishing bobs, mops, or buffs.

Polishing procedure:

1. Articles to be plated are polished with either calico or felt wheels dressed with 60-mesh emery.
2. A second polishing is given with wheels dressed with 90-mesh emery.
3. A third polish is given with wheels dressed with 120-mesh emery.
4. This is repeated a fourth time with 160-mesh emery.
5. A final polish is given with a suitable wheel dressed with the appropriate polishing compound (see Table V).

TABLE V. Polishing Compounds

	Tripoli	Crocus	Rouge	Chrome	White lime	Stainless steel
Alumina	25			20		
Calcium oxide				8	40	
Chromium sesquioxide				40		
Ferric oxide		75				
Magnesium oxide					25	
Paraffin wax		4				3
Rouge			80			
Silica	45	5		3		65
Stearic acid	14	15	9	27	19	15
Tallow			10		15	15
Triethanolamine		1	1	2	1	2
Metals for which the above compounds may be used	Brass, copper	Silver, steel, copper	Brass, nickel, silver, copper, gold	Chrome plate	Nickel, chrome, and silver plate	Stainless steel

Polishing methods for various types of objects:

1. Nickel-plated articles should be polished with an unbleached mop dressed with white lime composition.

2. Stainless steel is best polished with a felt bob and 80-mesh emery, continuing through the finer grades and finishing with stainless steel polishing compound.

3. Chrome plate is polished with a brown mop and special chrome polish or a white lime composition.

4. Brass castings are polished with a felt bob, dressed first with 90-grade emery followed by 120- and 180-mesh emery and finished with rouge.

5. Nickel silver and nickel are polished first with 180-mesh emery and then with tripoli, and finally finished with a soft rag mop dressed with white lime composition.

Chemical Cleaning. Chemical cleaning is carried out prior to any metal plating. Without the most scrupulous cleaning it is impossible to produce a finished coating which will resist corrosion and abrasion. Any grease or oil left on the metal surface of the article to be plated should be removed with the following hot caustic solution.

Degreasing Solution

Caustic soda	3 lb.
Soap	¼ lb.
Water	1 gal.

This is used at a temperature of 100° C. and the article to be degreased is immersed for about 5 minutes in this solution.

Scouring of Metal Surfaces. After the article has been degreased it is scoured to remove any lingering remains of rust or oxide on the surface. For scouring hard metals such as iron or steel, brushes with stiff bristles are used, whereas for softer metals such as brass or copper, soft bristled brushes are used. The brushes are dipped in water, the excess water is shaken out, and then the brush is dipped into a scouring compound.

Scouring Compounds

(*a*) For hard metals	
Powdered pumice (200-mesh)	1 lb.
Soap powder	2 oz.
Vienna lime powder	2 oz.
Potassium cyanide powder	1 oz.
(*b*) For soft metals	
Lime	1 lb.
Soap powder	8 oz.
Potassium cyanide powder	1 oz.

Washing. After each cleaning operation it is necessary to thoroughly wash the article. If cold water is used for washing, it is advisable to have the water running continuously at a rate at which the water is completely changed at least six times an hour.

Dipping. The acids used in pickling are effective in removing oxide scale, but they do not leave the bright surface desired as a finish. Thus a further acid dip is required to impart a truly bright surface. These dips are called scaling and bright, respectively; although the chemical components are the same in either dip, the proportions differ sufficiently to effect a marked difference in the finished product. Three acids, sulfuric, nitric, and hydrochloric, are used and in a precise proportion.

Sulfuric acid acts to reduce the attack of nitric acid on the metal and in this way maintains a smoother surface than could be achieved by nitric acid alone. Hydrochloric acid maintains the solution of both constituents of brass to the same degree. Nitric acid by itself attacks the copper, leaving a dull whitish surface. Hydrochloric in the proper amount prevents this happening, while an excess of hydrochloric acid dissolves the zinc and leaves an excess of copper on the surface. From this it may be seen that it is important to have the proper proportions of each acid.

	Scaling Dip	
Sulfuric acid	136 fl. oz.	1.84 sp. gr.
Nitric acid	26 fl. oz.	1.38
Hydrochloric acid	2 fl. oz.	1.17
Water	1 gal.	

Bright Dipping. To give a bright finish to the article, it should be quickly dipped in a solution composed of equal parts of nitric and sulfuric acid and then thoroughly washed to remove all trace of the acid.

Scratch Brushing. This step may be eliminated unless a special finish is desired on the work, i.e., a bright silken finish is obtained by scratch brushing the article with fine wire wheels or hard brushes. A fine frosted finish on gold and silver is also obtained by scratch brushing.

Plating Solutions

Among the many formulas used for plating different metals, the following are commonly used and produce satisfactory platings.

Copper Plating (acid).

Copper Sulfate	32 oz.
Sulfuric acid	6 oz.
Distilled water to	1 gal.
Temperature of solution	20 °C.
Voltage	3 volts
Current density	25–30 amp.

The *anodes* should be made of 99.5 % pure copper and the total surface area of the anode should equal the surface of the article being plated.

When rapid plating or thick deposits are required, the current density may be brought up to 300 amp. per square foot of surface on the article being plated, provided the solution is agitated during the plating.

Difficulties experienced during plating can be observed and corrected for by Table VI.

TABLE VI. Difficulties Experienced in Copper Plating.

End result	Cause	Remedy
Dark powder deposit	Excessive current	Reduce current
Pitted rough surface	Excessive acid	Analyze and add copper carbonate as necessary
Anode very bright	Excessive acid	Analyze and add copper carbonate as necessary
Plating too slow	Insufficient acid	Add sulfuric acid or copper sulfate as required

Cyanide or Alkaline Copper Plating.

Copper cyanide	3 oz.
Sodium cyanide	4 oz.
Sodium carbonate	2 oz.
Potassium hydrogen tartrate	8 oz.
Water to	1 gal.
Temperature of solution	30° C.
Voltage	3–4 volts
Current density	10–15 amp.

The anodes are of 99.5 % pure copper of surface area equal to that of the article being plated. See Table VII.

TABLE VII. Difficulties Experienced in Cyanide Copper Plating

End result	Cause	Remedy
Work blisters or tends to peel	Too much free cyanide	Analyze and add Cheveul's[1] salt as necessary
Anodes coated with a green slime	Not enough free cyanide	Analyze and add cyanide as necessary
Dark, red, or rough deposit	Current density too high	Adjust voltage and amperage
Bright and hard deposit	Current density too low	Adjust voltage and amperage
Work peels, but all operating conditions are correct	Work dirty	Remove plating and start over again with a thorough cleaning

[1] Chevreul's salt may be made as follows: 1.37 lb. of copper sulfate is dissolved in 1 gal. of warm water. 1.08 lb. of sodium sulfite is then dissolved in a second gallon of warm water. The two solutions are then mixed together by stirring. The resulting solution is allowed to settle for 18 hours and the supernatant liquid drained off. The remaining precipitate is washed six times with water; after each washing the precipitate is allowed to settle and the supernatant liquid drained off.

Nickel Plating.

Nickel ammonium sulfate	8 oz.
Nickel sulfate	4 oz.
Boric acid	2 oz.
Sodium chloride	2 oz.
Water to	1 gal.
Ph	5.8
Voltage	2.5 volts
Current density	6–8 amp./ft.

Cast or sheet *anodes* are of pure nickel. The ratio of anode to cathode surface is 1.1 : 1. See Table VIII.

TABLE VIII. Difficulties Experienced in Nickel Plating

End result	Cause	Remedy
No deposit	Current density too low, or bad connections	Adjust Variac Clean and tighten connections
Burned surface work rough or dull	Too high current density	Adjust Variac
No deposit	Too high current density	Adjust Variac
Deposit peels	Poor cleaning	Reclean work
	Omission of acid dip	
	Bath too acid	Analyze and adjust
	Bath too alkali	Analyze and adjust
Pitted deposit	Bath too acid	Analyze and adjust
	Impurities suspended in bath	Filter solution

Chromium Plating.

Chromic acid, 99.5 % CrO_3	55 oz.
Sulfuric acid	0.55 oz.
Water	to 1 gal.
Temperature of solution	35° C.
Voltage	4 volts
Current density	75–120 amp/ft^2.

The *anode* should be made of hard antimonial lead with an area at least 25 % greater than the area to be plated.

Specification of anode

Lead	93 %
Antimony	6 %
Iron	0.3 %
Copper	0.2 %
Bismuth	0.2 %
Arsenic	0.05 %
Manganese	0.05 %

The ratio of sulfuric acid to chromic acid should not exceed 1 : 100 for satisfactory plating.

Note. Except in very special cases, articles that are to be chrome-plated should be given a heavy coating of nickel first.

The older type of chrome plating where the plating is done directly on the basic metal without an undercoat of nickel is called *hard chrome plating.*

Chromic acid	35–55 oz.
Sulfuric acid	0.2–0.7 oz.
Water	to 1 gal.
Temperature of solution	60–75° C.
Voltage	4 volts
Current density	200–300 amp./ft^2.

The *anode* is the same as that for standard chrome plating.

Preparatory to hard chrome plating it is necessary to severely etch the surface of the basic metal in an anodic sulfuric acid etch. See Table IX.

TABLE IX. Difficulties Experienced in Chromium Plating

End result	Cause	Remedy
Blistering	Inferior nickel undercoat	Strip plating off and begin over again
Patchy deposit	Dirty anode connection	Clean connection
Burnt or dull deposit	Too low a temperature	Check and correct
Burnt deposit	Too great a current density	Adjust Variac
Dull patches of plating	Grease on the nickel undercoat	Strip plating and begin over again

Cadmium Plating. A definite advantage of cadmium plating over other metals is that it forms an alloy with a ferrous base metal. As a result, should the plating be scratched the article will not rust. Cadmium plating, however, is not suitable for nonferrous metals.

Cadmium oxide	3 oz.
Sodium cyanide	13 oz.
Water	1 gal.
Temperature of solution	20° C.
pH	13.9
Voltage	2 volts
Current density	25 amp./ft^2.

The *anodes* should be made of 99.0 % pure cadmium, with the impurity limits as follows:

Zinc	0.2 %
Lead	0.3 %
Copper	0.2 %
Antimony	0.1 %
Silver	0.2 %

The surface of the anode should be at least equal to the surface of the article being plated. When the bath is not being used the anode should be removed, thoroughly washed, and stored until needed again. See Table X.

TABLE X. Difficulties Experienced in Cadmium Plating

End result	Cause	Remedy
Deposit a dark color	Too high a current density	Adjust Variac
	Insufficient cyanide	Analyze for free cyanide and adjust
Deposit blisters	Too low metal content Too much free cyanide	Test and correct by the addition of cadmium oxide
Thin deposit	Low metal content	Analyze and adjust

Gold Plating.

Sodium gold cyanide	1 oz.
Sodium cyanide	2 oz.
Water to	1 gal.
Temperature of solution	Room temperature
Voltage	2.5–3.0 volts
Current density	As low as possible

The *anodes* are pure gold, 99.9 %, copper and silver not to exceed 0.01 %. The surface should approximately equal that of the article being plated. See Table XI.

TABLE XI. Difficulties Experienced in Gold Plating

End result	Cause	Remedy
Color of plating pale	Excess of free cyanide	Boil the solution and then make up to the correct volume with water
Deposit reddish brown in color and loose	Too little cyanide Bath temperature too low	Test and correct
Slow deposit and plating a copper color	Metal content too low	Analyze and correct

Gold wash. A gold plating may be obtained by a simple immersion, with the following solution.

Gold chloride	1 oz.
Potassium bicarbonate	32 oz.
Water to	1 gal.

The temperature should be about 80° C. and immersion is for a suitable time to give the desired coating.

Silver Plating.

Silver cyanide	3.5 oz.
Potassium cyanide	7.0 oz.
Potassium carbonate	5.0 oz.
Water to	1 gal.
pH	10.2–11.0
Temperature	20° C.
Voltage	1–2 volts
Current density	15 amp./ft^2.

The *anodes* are pure silver, 99.99 %, with the surface area in excess of the article being plated. See Table XII.

TABLE XII. Difficulties Experienced in Silver Plating

End result	Cause	Remedy
Deposit blisters and peels on polishing	Bad cleaning	Strip plating and begin over again
	Bad solution	Analyze and correct solution
	Base metal not suitable for gold plating	Plate coating of copper on article
Deposit dark and spongy	Current density too high	Adjust Variac
Difficulty in maintaining the proper current density with the voltage	Lack of free cyanide	Add potassium cyanide

Lacquering. Few of the common metal finishes are immune to surface oxidation or discoloration, and in order to assure permanency common practice is to cover the metal with a thin film of lacquer. This is done in three different ways, dipping, spraying, or immersion, depending on the type of lacquer to be used and the article to be lacquered. See Table XIII.

TABLE XIII. Difficulties Experienced in Lacquering

End result	Cause	Remedy
Poor adhesion or rough film	Oil or grease on surface prior to lacquering	Remove lacquer and clean surface
Cloudy surface	The atmosphere too cool or humid	Raise room temperature or warm article
Rainbow colors	Lacquer too thin	Redo with heavier coat of lacquer
Spots or specks	Dust in the air	Remove the lacquer and do over again
Rippling	Drying too rapidly	Reduce drying temperature

XI

PURIFICATION OF MERCURY

Even minute traces of foreign metals cause striking changes in the physical properties of mercury and interfere with applications to scientific instruments. The purity required determines the type and thoroughness of cleaning required.

FILTRATION

The mercury may be freed of coarse particles of insoluble matter by flowing through a pinhole in a conical paper filter. Other materials such as leather,[1] bamboo,[2] silk,[3] chamois,[4] and fritted glass[5] have been used for this preliminary filtering.

CHEMICAL CLEANING

Meyer[6] reported on the use of an acid tower (Figure 1) which is filled with a solution of 10 % nitric acid, or better, mercurous nitrate, with the mercury allowed to pass through the solution in small droplets. The efficiency of this tower is improved if the modified tower developed by Friedrichs[7] is used. Wichers[5] reported on a method of agitating mercury in a dilute solution of nitric acid by blowing air through it (Figure 2).

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[1] Bolton, *Electrochem.*, **2,** 76 (1896).

[2] Karsten, *Instrumentenk*, **8,** 135 (1888).

[3] Patten, H. E., and Mains, S. H., *Ind. Eng. Chem.*, **9**, 600 (1917).

[4] Moor, C. J., *J. Am. Chem. Soc.*, **32**, 971 (1910).

[5] Wichers, E., *Chem. Eng. News*, **20**, 1111 (1942).

[6] Meyers, L., *Z. anal. Chem.* **2**, 241 (1863).

[7] Friedrichs, F., *Angew. Chem.*, **27**, 24 (1914).

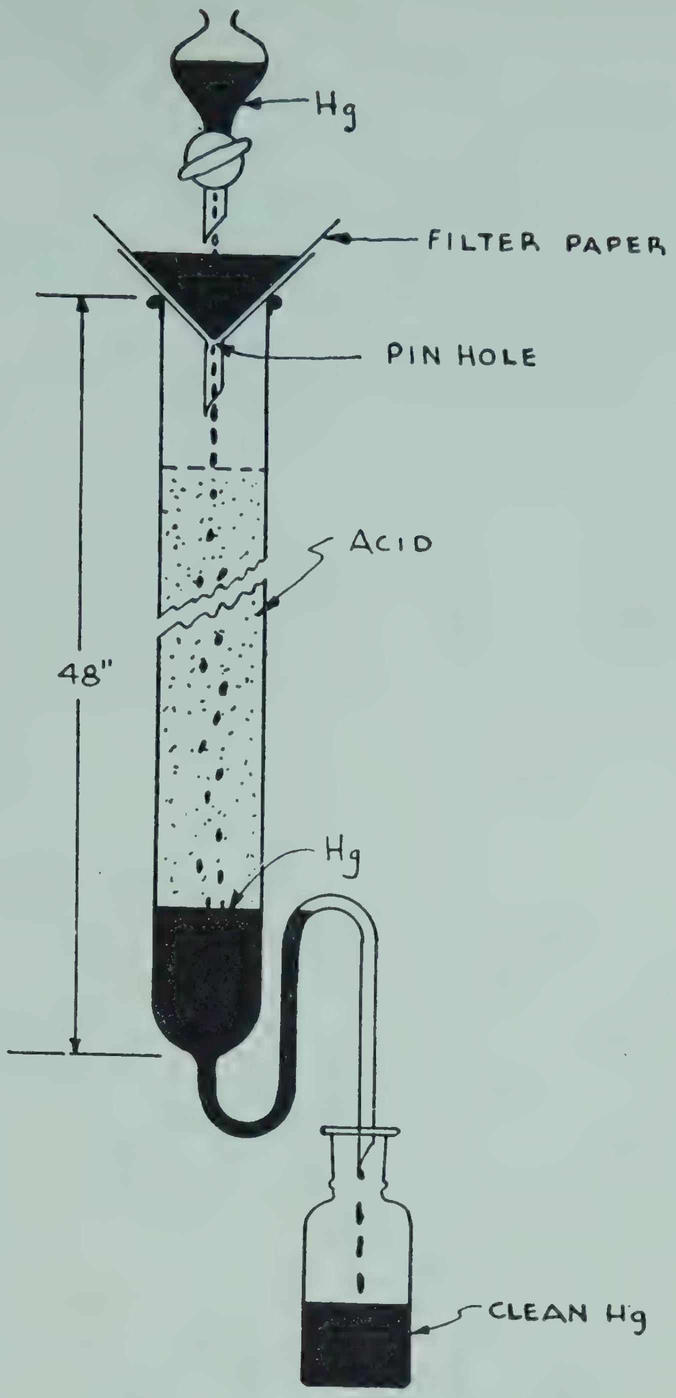
Hg
FILTER PAPER
PIN HOLE
48"
ACID
Hg
CLEAN Hg

Figure 1. Mercury acid wash tower.

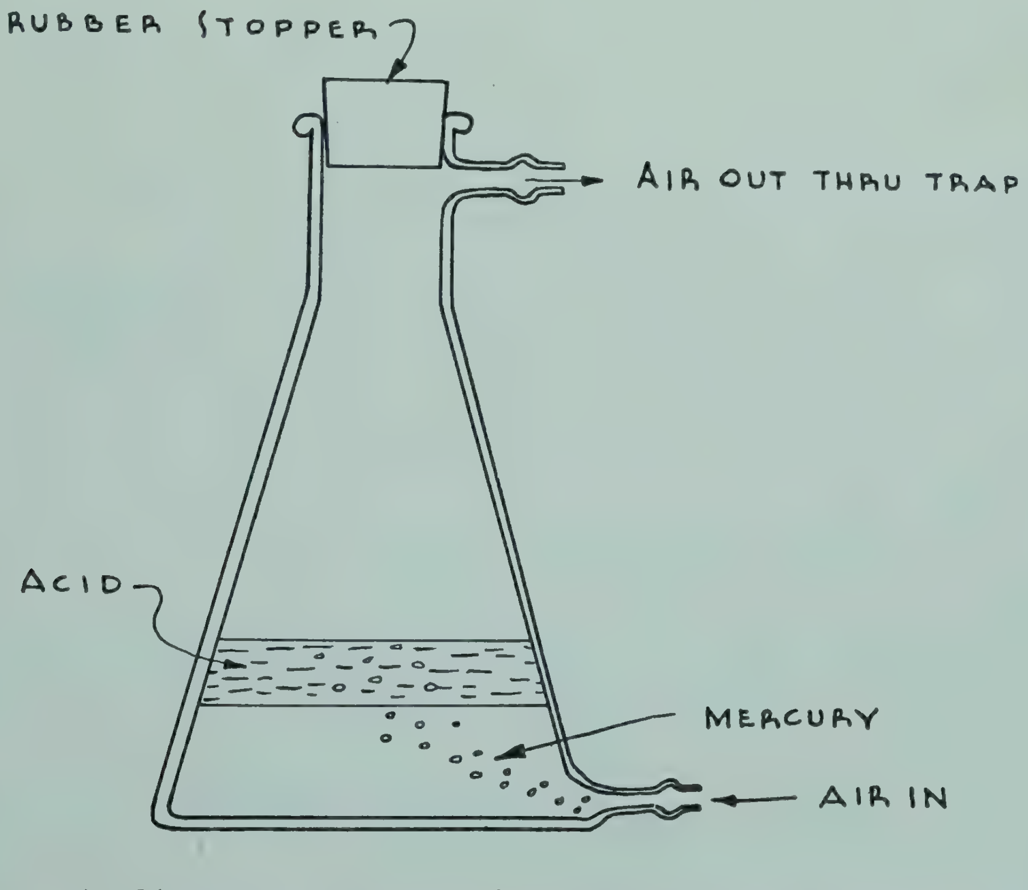
RUBBER STOPPER
AIR OUT THRU TRAP
ACID
MERCURY
AIR IN

Figure 2. Simple apparatus for agitating mercury in acid.

OXIDATION WITH AIR

The author[8] has described a metal pipe agitator for oxidizing the base metals in large amounts of mercury. This agitator is pictured in Figure 3 and is made from a 34-in. length of 4.5-in. pipe sealed off at one end with a flat steel disc fitted and welded in place. An air inlet comes in at the bottom and a baffle is fastened near the top to prevent splash during operation. A needle valve at the bottom drains the agitator at the end of the run. A No. 3860–15 BE Norton grinding wheel is cut down and mounted as shown in Figure 3. Miracle cement[9] is used to seal the periphery of the wheel to the side of the pipe, thus forcing the air to pass through the grinding wheel, which acts as a fritted bubbler. This apparatus requires no special attention and may be left running overnight when the mercury is especially dirty. The oxidized base metals are removed from the surface of the mercury by one of the filtration methods described.

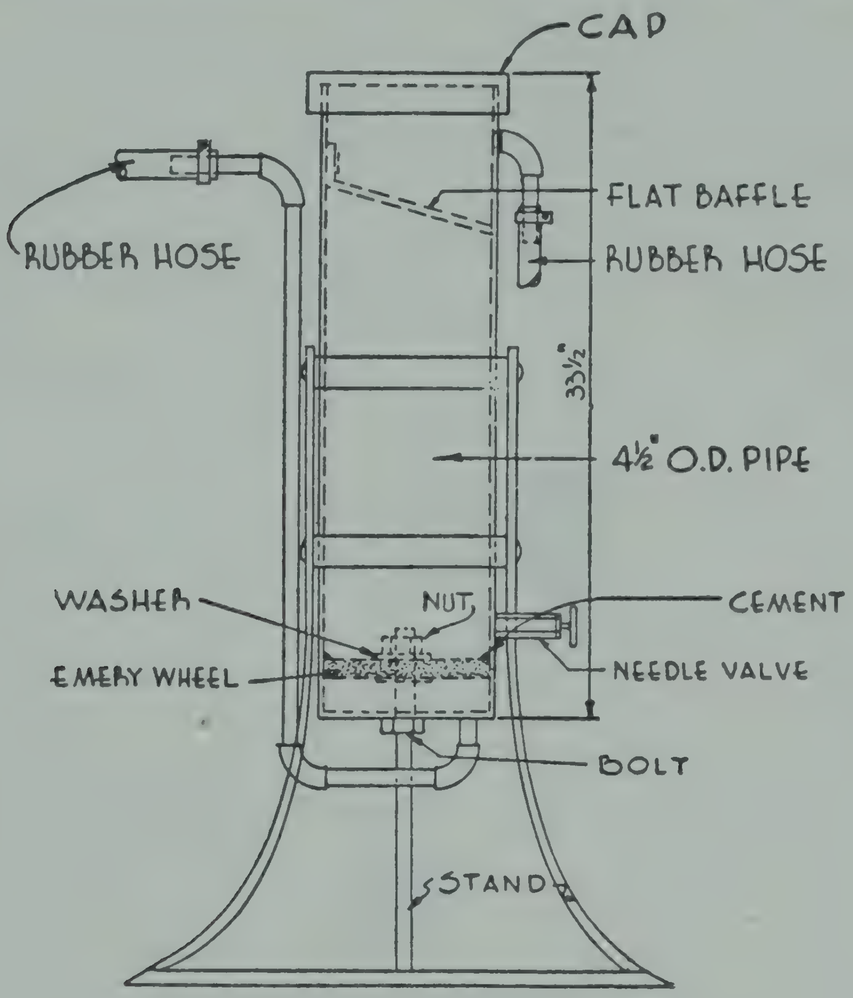

Figure 3. Metal pipe mercury agitator.

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[8] Wheeler, E. L., *Anal. Chem.*, **24**, 751 (1952).

[9] Miracle Adhesive Corp., Newark, New Yersey.

DISTILLATION

For many uses the methods of purification just described are sufficient; however, Hulett[10] has shown that the only way to remove impurities completely is with three distillations. According to Hulett when a little air is allowed to pass over with the mercury vapor during the distillation, most of the metallic impurities are oxidized while the mercury vapor is not affected. For the removal of platinum or gold, two distillations may be sufficient, but for silver, which is the most difficult to remove, three distillations are required.

Single Distillation

Figure 4 shows a simple still which may be constructed from materials found in any laboratory. Air is bubbled through the mercury by means of a fine capillary sealed to the bottom of a $ inner through joint.

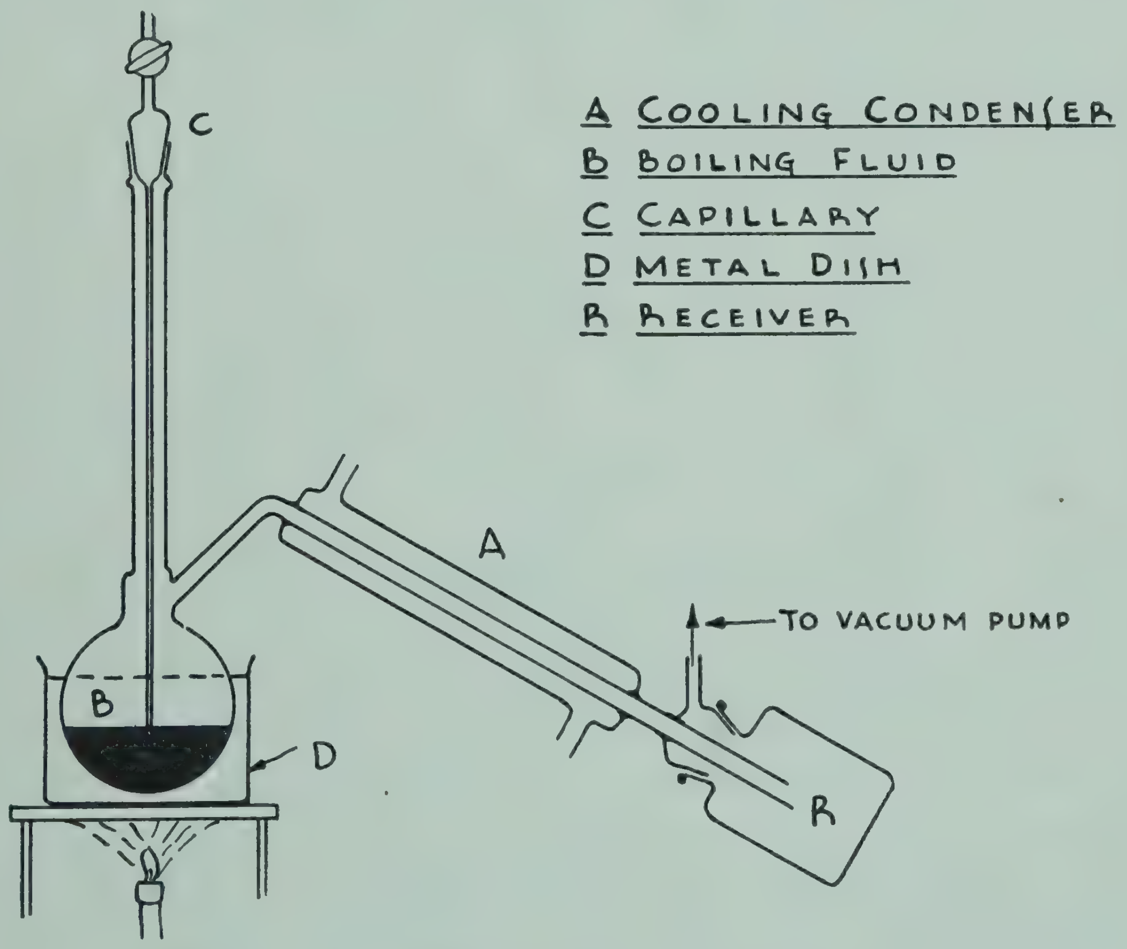

Figure 4. Single stage mercury still.

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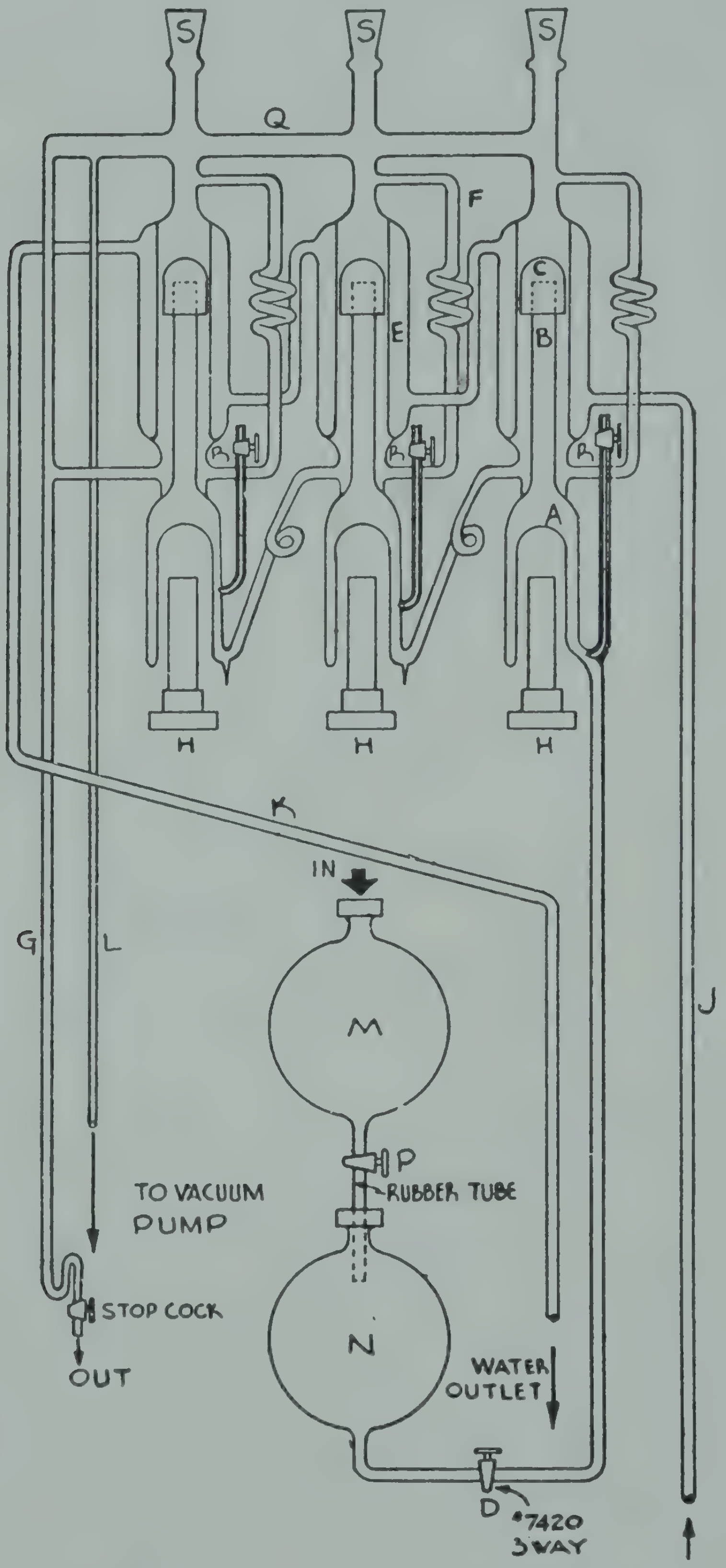
[10] Hulett, G. A., *Phys. Rev.*, **33** (1), 307 (1911).

Triple Distillation

Figure 5 shows an apparatus suitable for the triple distillation of mercury. Modified umbrella-type diffusion pumps were


S
S
S
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C
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R
A
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H
H
K
IN
G
L
M
J
P
RUBBER TUBE
TO VACUUM PUMP
STOP COCK
OUT
N
WATER OUTLET
D
#7420 3WAY

Figure 5. Triple mercury still.

chosen for this because of their high vapor throughput, and with a 350-watt heater for each boiler, triple distilled mercury can be taken off at a rate of 2 pounds an hour. In Figure 5 it is seen that the mercury vapor goes from the boiler *A* up through chimney *B* until it strikes umbrella *C*, which deflects it down onto wall *E* of the condenser. Here the vapor is condensed and the liquid mercury drops down the wall to the boiler through line *G*. *F* is an equalizing line to maintain pressure above and below the umbrella. The third unit return line, *G*, extends down 80 cm. and is the product take-off line. *H* is the boiler heater. *J* is the water inlet and *K* is the outlet from condenser *E*. *L* is the line to the vacuum forepump. *M* is the reservoir from which crude mercury is passed into flask *N* at a rate controlled by screw clamp *P*. In place of *P* a float valve described by Carlsen and Borchardt[11] may be used. By maintenance of a constant level in *N* the proper level is held in boiler *A*. Stopcock *D* may be eliminated if desired, but it is convenient for draining *N* and the boiler at the end of the distillation. Vacuum line *L* is connected to manifold *Q*, and thus to lines *F*, all units being held at the same pressure.

Air is allowed to enter each boiler through *R*. These stopcocks are Corning #7300 capillary stopcocks, and a smooth-fitting piece of Nichrome wire can be inserted in the capillary section above the stopcock to give the correct amount of air when the stopcocks are wide open. In place of this sliding-wire regulator $ joints may be sealed above the stopcock and a $ cap made with a fine capillary constriction of the proper size to allow the right amount of air to pass. The small capillary must be protected from becoming clogged with fine particles of dust from the atmosphere. The lines from *R* are also useful in draining the boilers and cleaning the still at the end of a distillation. Ground joints and caps at *S* are also useful during the cleaning operations.

Each boiler is served not only with its individual resistance heater, but also a switch and separate rheostat of 300-watt and 5.48 amp. capacity. With these controls the still can be run all day with very little attention other than an occasional adjustment of the boiler levels and rheostat. Slightly closer attention should be directed to flask *N* to maintain the proper level there, as the even operation of all the boilers is partly dependent on this.

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[11] Carlsen and Borchardt, *Ind. Eng. Chem., Anal. Ed.*, **10**, 94 (1938).

The pressure at which the still is run should be maintained between 20 to 25 mm., with the aid of any standard pressure regulator or manostat, and with the boilers at about 200° C. Under these conditions with enough air passing over with the vapor, removal of the base metals will be complete in the first distillation. However, in this still the operator has the choice of bubbling air in one or all three boilers as the situation warrants.

XII

FRACTIONAL DISTILLATION

Fractional distillation is one of the most important techniques in chemical research. Because of this, glassblowers in research laboratories have many requests to build distillation equipment to perform both simple and special distillation operations. The primary purpose of this discussion of fractional distillation is to bring to the glassblower a practical knowledge of distillation and distilling columns as an aid in designing and constructing necessary equipment.

For a detailed study of the theory of distillation, recommended reading is *Technique of Organic Chemistry,* Volume 4.[1] A book by Carney[2] will also be found informative.

TERMINOLOGY

The following terms are used in this chapter.

Column. The section of the fractionating assembly in which rectification takes place.

Still Head. The portion of the fractionating assembly above the rectifying section, usually consisting of one or more condensers and a reflux dividing device.

Still Pot. The flask or container in which the material to be distilled is heated.

Rectification. A distillation carried out in such a way that the vapors come in contact with the descending reflux, resulting in an exchange of heat and materials and thus enriching the vapor with the more volatile component.

Fractionation. The common term applied to rectification.

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[1] Interscience, New York, 1951.

[2] Carney, T. P., *Laboratory Fractional Distillation,* Macmillan, New York, 1949.

Rectifying Section. The section of the column occupied by the packing or rectifying devices.

Packing. Particles of material placed in the rectifying section to increase the liquid and vapor contacting surface (preferably inert).

Operating Holdup. The volume of liquid and vapor in the column under operating conditions.

Static Holdup. Sometimes referred to as operating loss. This is the volume of liquid held up in the column after the distillation is completed and the column drained.

Throughput. The total amount of material rising to the head of the column per unit time.

Reflux. The condensate that is returned to the column, flowing countercurrent to the vapor stream.

Vapor Velocity. The upward rate of vapor in the column.

Pressure Drop. The decrease in pressure between two sections of the column, usually considered between the still head and the still pot.

Reflux Ratio. The ratio between the amount of condensate returned to the column and that withdrawn at the head as product.

Partial Reflux. The process in which all of the condensate is returned to the column and the rest drawn off as product.

Total Reflux. The process in which all of the condensate is returned to the column and no product removed.

Perfect Plate. A section of the column which effects complete equilibrium between the vapor and the liquid.

Plate Efficiency. The degree with which a section of column approaches the vapor-liquid equilibrium of a perfect plate.

Theoretical Plate. A mathematical unit of efficiency in the operation of a fractionating column.

H.E.T.P. The Height Equivalent to a Theoretical Plate, that is, the length of a column necessary to make the same separation as is made by one perfect plate.

Total Condenser. A condenser which condenses all of the vapors and returns part of the liquid to the column as reflux, directing the remainder to the product take-off line.

Partial Condenser. A condenser in which part of the vapor is condensed and returned to the column as reflux, while the remainder is condensed separately and removed as product.

Fractionating Assembly. The fractionating column and all of its accessories.

Binary Test Mixture. A two-component mixture that is used to determine the separating efficiency of a column.

Simple Distillation. Future reference to simple distillation in this chapter is to a distillation in which a mixture of two miscible liquids is heated. The vapors thus involved will usually be richer in the lower boiling component than was the original mixture. When these vapors are condensed and removed, the condensate will contain a larger proportion of the lower boiling component and the remaining liquid, more of the higher boiling material.

METHODS OF DISTILLATION

There are two methods of distillation used: continuous or batch.

Continuous Distillations

A continuous feed of preheated material of a constant composition is admitted at a point near the middle of the column; at the same time the simultaneous removal of several fractions from different sections of the column is permitted. Each fraction, although different from the others, has a fairly constant composition. The lower boiling fractions are removed at the head of the column and the higher boiling fractions are taken from the bottom.

Batch Distillations

An initial charge is placed in the still pot and product is removed only at the head. This provides for a progressive series of fractions, each having a different composition. This method is more satisfactory for analytical distillations and will be considered more fully.

FACTORS TO BE CONSIDERED IN DESIGNING DISTILLATION EQUIPMENT

The design of columns, still heads, and kettles for general laboratory distillations depends largely on the following four points.

Efficiency of Separation Desired

In choosing a column to perform a given separation, it is desirable to know the approximate number of plates required to effect the desired separation. Carney[2] (pages 28 to 35) gives the formula for estimating the plates required for a given separation. The efficiency of separation is determined by the number of theoretical plates a column possesses at an infinite reflux ratio. This is merely a comparison between stills and does not necessarily represent the efficiency under actual operating conditions. We can, however, safely assume that with two columns the one showing the highest theoretical plate value at total reflux will probably have the most theoretical plates under operating conditions. Other considerations being equal, the rectifying column showing the greatest enrichment per unit length is the most desirable.

Rate of Distillation

With any given column there is an optimum rate, above which there will be poor separation, and below which the increased separation will not warrant the extra time required. Naturally the ideal column would have a large number of theoretical plates, small holdup, low pressure drop, and a high throughput. Actually it is necessary to compromise to obtain the maximum efficiency. Usual distillation procedure is to keep the distillation rate constant. Although some operators increase the rate when collecting a pure component and decrease the rate between components. According to Podbielniak,[3] distillations may be run in less time using this procedure with no loss in accuracy.

[3] Podbielniak, W. J., *Ind. Eng. Chem., Anal. Ed.*, **5**, 176 (1933).

Temperature Range To Be Covered

The temperature range over which the still will be operated will partly determine the design of the still. When the temperatures of the distillations are high, precautions must be taken to prevent, or compensate for, excessive heat loss. If this is neglected, fluctuation in the reflux results, causing flooding in the column or lack of reflux at the still head. A silver-plated, vacuum-jacketed system up to the reflux condenser is the best arrangement for solving this problem. (See p. 241.)

Material To Be Fractionated

This is of importance in designing a column because of such physical and chemical properties as viscosity, surface tension, latent heat, tendency to foam, and corrosiveness. These factors also determine, in part, the type of packing to be used in the finished column.

CONSTRUCTIONAL FEATURES

The following construction features should be considered in designing an efficient fractionating unit.

Still Pot

The still pot should be a corrosion-resistant kettle of the proper size for the characteristics of the charge being distilled. For example, it should be large enough to care for thermal expansion and possible foaming of the material being distilled, but it should not be so large that the evaporating surface of the charge is great enough to cause flooding in the column before brisk boiling begins. The still pot should be well insulated and evenly heated by an electrical heating unit that can be closely controlled.

Column

According to Perry[4] the ratio of the height of the column to its diameter should not be less than 15 to 1 to prevent undue channeling. It should be well insulated and, in some cases, have a closely regulated source of external heat to compensate for radiated or lost heat.

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[4] Perry, *Chemical Engineers' Handbook*, p. 1195. McGraw-Hill, New York, 1934.

Packing

The choice of packing depends on the intended use of the still. For viscous materials, those that entrain badly, or those that foam, glass cylinders perform well. For atmospheric and some reduced pressure distillations, single-turn helices of Pyrex brand glass give good results for many materials. The packing should be corrosion-resistant and have a large surface area per unit volume. A small holdup and pressure drop are also desirable qualities. In addition, it should have enough free space for a satisfactory throughput. Baker, Theodore, Chilton, and Vernon[5] have noted that the tendency of a packed column to channel will be less if the ratio of the column diameter to the diameter of the individual packing units is greater than 8 to 1.

Still head

The function of the still head is to receive the vapor from the top of the column, condense part or all of it, and return liquid reflux to the column. Generally, no fractionation takes place in the still head. If, however, it includes a partial condenser, some fractionation does take place. A still head, like the column, should be properly insulated and designed for the temperature and material to be distilled. When an immersion thermometer is used, the thermometer well should be located so as to connect to or be part of the vapor path below the condenser. The thermometer well should be insulated and vacuum-jacketed, if possible. For high-temperature distillations it will be necessary to have the vapor path completely vacuum-jacketed up to and above the take-off line, thus insuring that no condensation from heat loss takes place between this point and the top of the column. Columns with the head built into the vacuum jacket directly above the rectifying section are especially suitable for high-temperature distillations.

[5] Baker, Theodore, Chilton and Vernon, *Trans. Am. Inst. Chem. Engrs.*, **31**, 302 (935).

THERMOMETER

An accurate knowledge of the boiling point of the distillate is of primary importance when a known compound is being purified or a new compound is being distilled. It is poor economy to use cheap, unreliable thermometers at the still head. The still pot temperature is also important in enabling the operator to predict cuts and prevent superheating. This kettle temperature, however, need not be known as accurately as the still head temperatures.

LAGGING OR INSULATION

There are many variations of jackets for columns, the most common being asbestos, magnesia, air jacket, vacuum jacket, and electrical heating mantles. The relative efficiency of lagged and unlagged columns was determined by Murshal and Southerland,[6] who found that a properly lagged Hempel column was from 2.4 to 41.5 times as efficient as an unlagged column operating at the same distillation rate.

Silvered vacuum jackets provide the most efficient insulation. In the case of micro columns, columns used for reduced-pressure distillations, or columns used for high temperatures (up to 400° C.), electric heating mantles[7] over the vacuum jacket give the optimum in results. With this combination jacket, fine control of the electric heating mantle is not necessary. Maintaining the mantle within 5 ° to 10° C. of the temperature inside the column effectively reduces the heat loss through the vacuum jacket.

Actual experiments with 27 mm. O.D. (1.05-in.) columns under comparable conditions show the rate of decrease in temperature of a liquid in the column from 210° to 200° C. to be as follows:

Insulation	Rate of Decrease in Temp. °C./min.	Relative Rates
Silvered and vacuum-jacketed	0.36	1
85 % magnesia, 15 % asbestos by 0.88 in. thick	2.5	7
None	14.0	38.8

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[6] Murshall, M. J., and Southerland, B. P., *Ind. Eng. Chem.*, **19**, 735 (1927).

[7] Glass Col Apparatus Co., Terre Haute, Indiana.

If magnesia lagging is to be used, it should be of the same thickness as the diameter of the column. Thus a 25 mm. O.D. column should have 25 mm. of lagging and a 100 mm. O.D. column should have 100 mm. of lagging. Heat transmission through cylindrical lagging on a cylindrical pipe varies inversely as the logarithm of the ratio between the external and internal diameters of the lagging. Assuming heat conductivity to be a fixed constant at a given temperature, a 25-mm. column covered with 25 mm. of lagging should lose heat at exactly the same rate as a 100-mm. column insulated by 100 mm. of lagging. Using a thicker lagging does not materially reduce heat loss because, by the logarithmic formula, if the insulation is twice as thick as the diameter of the column, the heat loss will be reduced only by an additional 20 %.

The construction of columns with electrical heat-compensating jackets is relatively simple, and columns of this design are particularly adapted for laboratories where glassblowing facilities and the equipment for evacuating columns are not available. The best temperature control is obtained when the heater is wound in several sections, with the individual sections controlled independently by a variable transformer. The recommended maximum length for each section is 3 ft. Figure 1A gives a cross section of

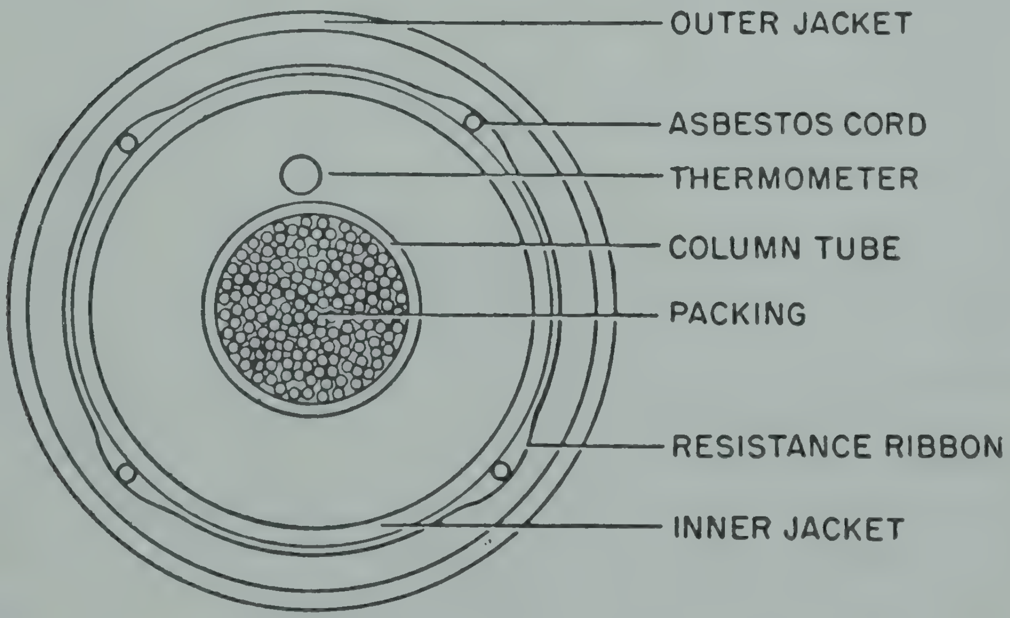

Figure 1A. Cross section of electric heating jacket. (From A. L. Glasebrook and F. E. Williams in *Technique of Organic Chemistry*, Vol. 4, Interscience, New York, 1951.)

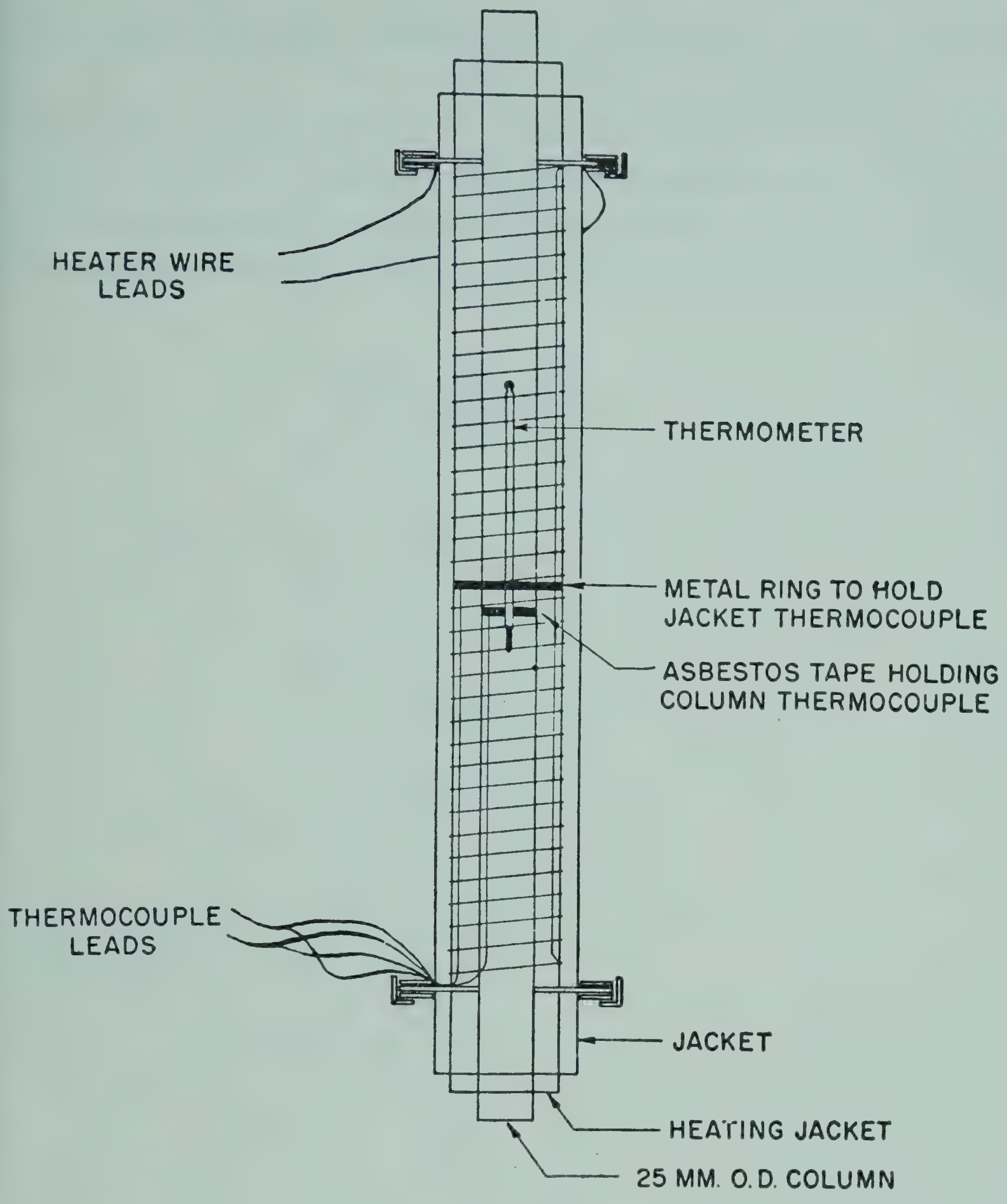
HEATER WIRE LEADS
THERMOMETER
METAL RING TO HOLD JACKET THERMOCOUPLE
ASBESTOS TAPE HOLDING COLUMN THERMOCOUPLE
THERMOCOUPLE LEADS
JACKET
HEATING JACKET
25 MM. O.D. COLUMN

Figure 1B. Electric heat compensating jacket. (From A. L. Glasebrook and F. E. Williams in *Technique of Organic Chemistry*, Vol. 4, Interscience, New York, 1951.)

a column with a compensating heating jacket and Figure 1B shows a vertical view of the heater. The tube wound with the resistance heating wire should be about ¾ in. larger than the outside diameter of the column and slightly longer than the section to be packed. Three or four lengths of asbestos cloth tape should be fastened longitudinally on the jacket to prevent the hot resistance wire from coming into contact with the glass, and the spacing of the wire coil should be ½ to ¾ in. The asbestos tape also prevents the wire loops from slipping together and creating a direct short.

The ends of the heater section are wound with several layers of asbestos tape, and a second glass tube of suitable diameter to fit snugly over the asbestos tape is fitted over the jacket to protect the heater from cool drafts of air and to preserve full visibility of the packed section. In place of the rolled asbestos tape separators, Transite plates may be cut to fit snugly between the column and separate members of the compensating jacket.

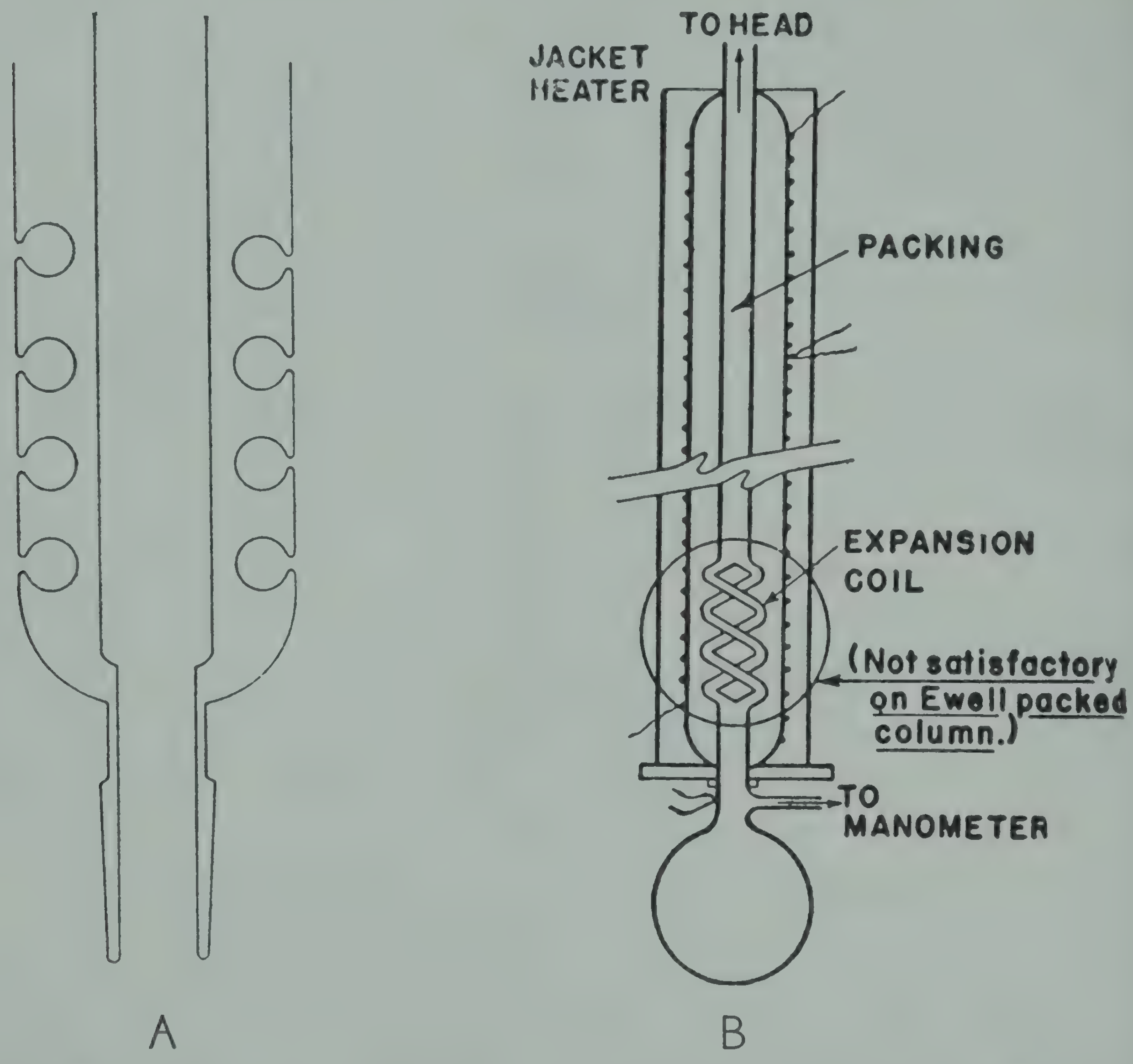
TO HEAD
JACKET HEATER
PACKING
EXPANSION COIL
(Not satisfactory on Ewell packed column.)
TO MANOMETER
A
B

Figure 2. Expansion sections for vacuum-jacketed columns. (From A. L. Glasebrook and F. E. Williams in *Technique of Organic Chemistry*, Vol. 4, Interscience, New York, 1951).

One thermometer may be used as shown in Figure 1 in conjunction with several thermocouples, or several thermometers may be spaced at different levels in the jacket.

When silvered vacuum jackets are used, some means of compensating for the difference in temperatures between the inner tube and the outer jacket must be used to avoid undue stress on the glass seals and the resulting breakage. External expansion bellows can be formed on the outside jacket as a means of compensating for the differential expansion, or an internal coil may be sealed on the lower end of the inner tube (Figure 2).

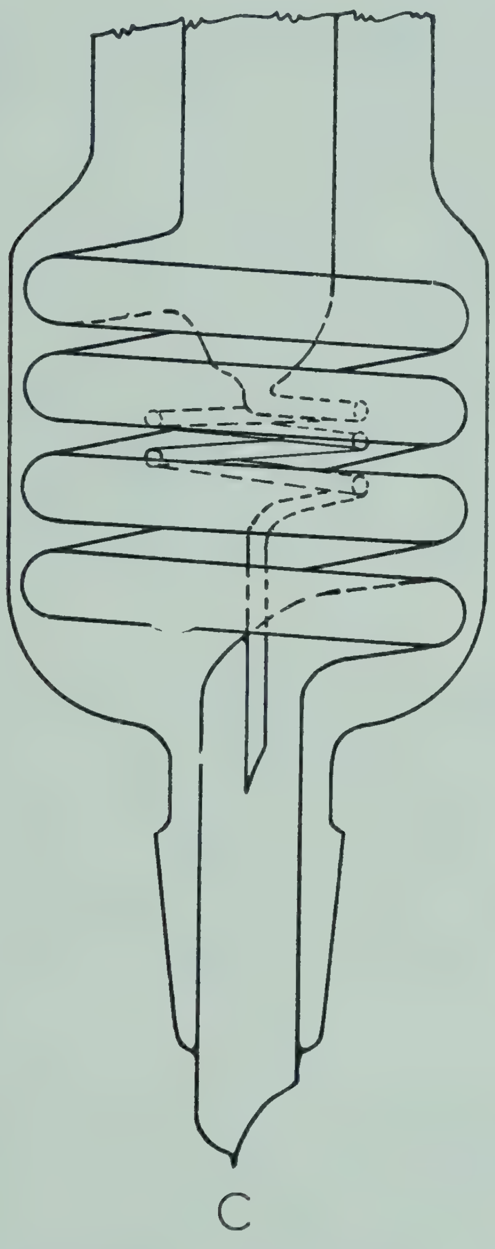

Figure 2. (Continued).

COLUMN PACKING

The ideal packing is one that has a large surface for contact between the liquid and the vapor. It should pack evenly in the column, thus assuring uniformly distributed interstices, or free space, and have enough free space for the required throughput. The irregularity of the interstices in any packed column is a complicating factor, making experimental values necessary in evaluating or comparing packings. Fenske, Tongberg, and Quiggle[8] noted that the surface area per cubic inch of packing, multiplied by the percentage of free space, gave numerical ratings which paralleled the usefulness of various packing materials.

Glass Cylinders

These are more rugged and easier to make than any other packing. Glass cylinders 3 mm. long and 5 mm. O.D. with 1-mm. walls are satisfactory for most purposes. Tests with a mixture of carbon tetrachloride and benzene give the H.E.T.P. of this packing as about 3.5 in. in a 1-ft. column of 22 mm. I.D. The maximum rate of take-off for liquids boiling around 200° C. and maintaining a reflux of 90 % is about 3 ml. per minute. When greater throughput is required, columns packed with rings 4 mm. long by 10 mm. O.D. with 1.5-mm. walls may be used. The H.E.T.P. of such rings in a column of 25 mm. I.D. is 6 to 7 in.

Raschig Rings

These are small thin-walled cylindrical tubes of earthenware, glass, metal, or other inert material, and they are usually made with equal height and diameter. Owing to their uniform shape and dimensions, they pack in a column evenly and present a reasonably large surface area and free space.

Solid Packing

Solid-type packing includes glass beads and balls of borosilicate glass, particles of stone, Carborundum, and other inert material. These were the earlier types of packings developed and are still used in simple laboratory fractionating units. Solid-type packing offers a relatively small surface area per unit column and has a tendency to flood at a low boil-up rate.

Spiral Packing

A spiral coil of wire which fits closely into the column makes an efficient packing. Glass spiral may be substituted for the metal with about the same efficiency. The fragility of glass spirals, plus the difficulty of getting a glass spiral to fit closely to the walls of the tube, makes glass spiral packing less widely used than wire packing. Podbielniak[9] describes a 3.8-mm. column packed with No. 20 wire wound with 6 to 7 turns per inch. The Todd Scientific Co.[10] manufactures a column with a metal spiral packing. This column actually consists of three interchangeable rectifying sections and one electrically heated jacket. The smallest section is 5 mm. I.D. and has the metal spiral packing. The other two

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[8] Fenske, M. R., Tongberg, C. O., and Quiggle, D., *Ind. Eng. Chem.*, **26,** 1169 (1934).

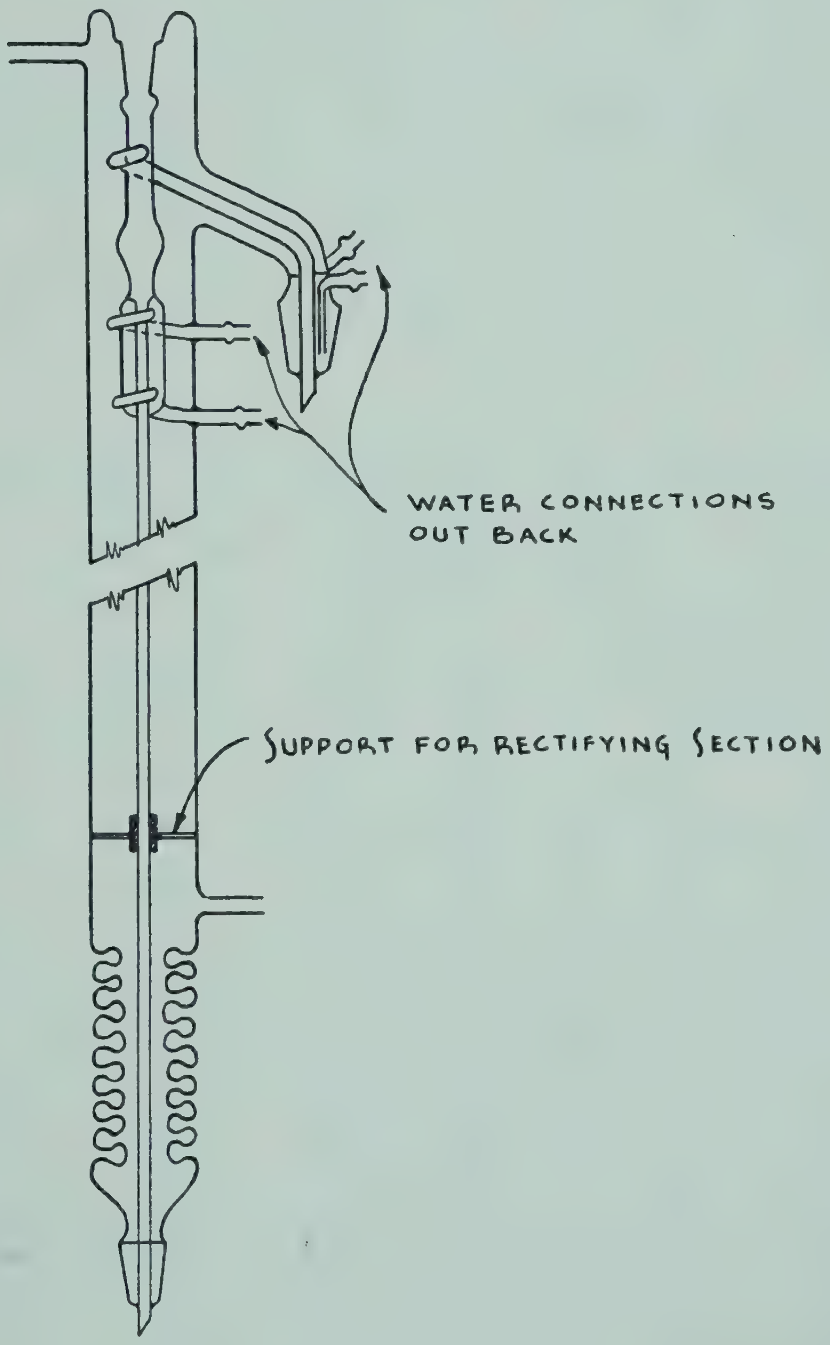

Figure 3. Fractionating column with wire spiral packing.

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[9] Podbielniak, W. J., *Ind. Eng. Chem. Anal. Ed.*, **3**, 179 (1937).

[10] 160 State Street, Springfield, Pennsylvania.

sections, 12 mm. I.D. and 25. mm. I.D., are packed with 4-mm. diameter glass helices. By interchanging these three rectifying sections, volumes of liquid from 2 ml. to 5000 ml. may be fractionated. A vacuum-jacketed vapor take-off column with wire spiral packing is shown in Figure 3.

Ground Glass Fused to the Inner Wall of the Rectifying Tube

This gives about the same efficiency as the glass spirals in micro column, without the danger of frequent breakage experienced with glass coils. In making the ground-glass-coated tubing, a tube of the desired size is half-filled with ground glass (50- to 60-mesh). One end of the tube is rotated in a gas-oxygen flame until a layer of ground glass has fused to the side wall, and the heating is continued down the length of the tubing until the desired rectifying section has been prepared. The tube is then lifted to a vertical position and the excess glass removed by gently tapping on the side of the tube.

TABLE I. Operating Characteristics of Metal Helices *

Column							
Length, cm.	Diam., cm.	Throughput, ml./min.	Theor. plates	H.E.T.P., cm.	Holdup, ml./plate	Effic. factor	Pressure drop, m.m. Hg/plate
$^1/_{16}$-In. I D. stainless steel of No. 34 wire							
71	0.8	0.38	55	1.3	0.16	140	—
105	2.54	8.3	65	1.6	1.2	420	0.02
105	2.54	16.7	62	1.7	1.6	630	0.07
105	2.54	51.0	43	2.5	3.3	925	0.65
$^3/_{32}$-In. I.D. stainless steel of No. 20 wire							
70	0.8	0.54	27	2.6	0.29	110	—
81	1.2	7.5	37	2.2	0.64	820	—
266	2.54	8.5	140	1.9	1.2	525	0.13
266	2.54	16.9	126	2.1	1.6	630	0.05
266	2.54	50.7	88	3.0	3.4	900	0.57
225	5.08	22	58	3.9	13.5	98	0.009
225	5.08	44	54	4.2	16.3	162	0.048
225	5.08	132	44	5.2	31.0	255	0.72

*From A. L. Glasebrook and F. E. Williams in *Technique of Organic Chemistry*, Vol. 4, Interscience, New York, 1951.

Metal Helices

Metal helices were developed by Fenske, Tongberg, and Lawroski.[11] Of the many types of packings developed, probably the most widely used are single-turn helices of either metal or glass. Metal helices are commercially available in standard size of $^{3}/_{64}$, $^{1}/_{16}$, $^{3}/_{32}$, and $^{5}/_{32}$ in., allowing for both large and small columns to be packed with the proper size for the ratio between

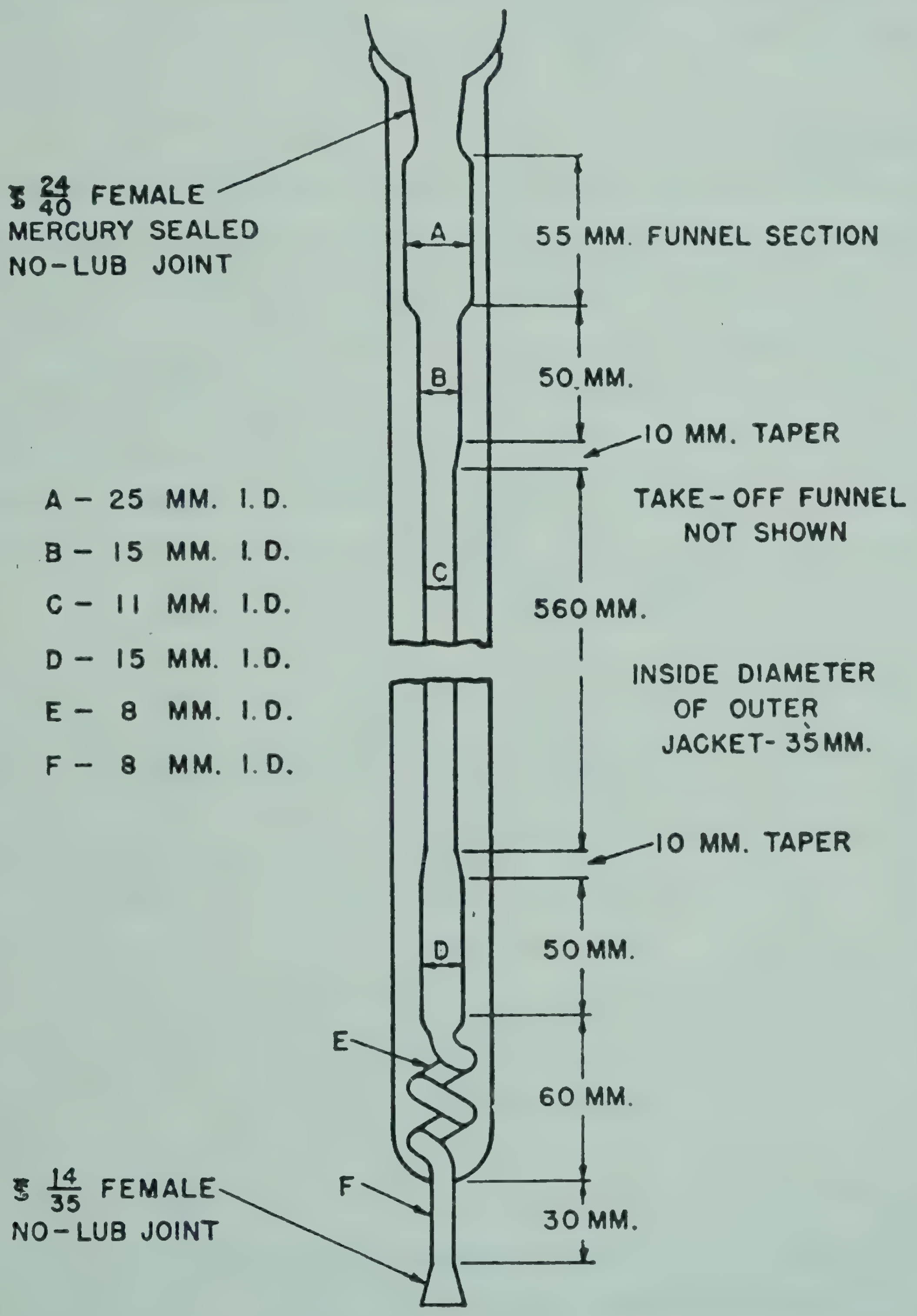
$ 24/40 FEMALE
MERCURY SEALED
NO-LUB JOINT
A
55 MM. FUNNEL SECTION
B
50 MM.
10 MM. TAPER
TAKE-OFF FUNNEL
NOT SHOWN
A - 25 MM. I.D.
B - 15 MM. I.D.
C - 11 MM. I.D.
D - 15 MM. I.D.
E - 8 MM. I.D.
F - 8 MM. I.D.
C
560 MM.
INSIDE DIAMETER
OF OUTER
JACKET- 35 MM.
10 MM. TAPER
D
50 MM.
E
60 MM.
$ 14/35 FEMALE
NO-LUB JOINT
F
30 MM.

Figure 4. Column for analytical distillations.

[1] Fenske, M. R., Lawroski, S., and Tongberg, C. O., *Ind. Eng. Chem.*, **29,** 957 (1937).

column and packing. In addition to being made in different sizes, metal helices are made from different sizes of wire. Metal helices have achieved their popularity as packing for distillation columns mainly because of their high efficiency and relatively low cost. Table I gives the operating characteristics of metal helices. The vacuum-jacketed, 80 plate analytical column shown in Figure 4 is packed with $^3/_{64}$ in. metal helices and has a rated H.E.T.P. of 0.35 in. The maximum boil-up rate is about 2.5 ml. per minute and the operating holdup is 8 ml.

Glass Helices

Glass helices were developed as a substitute for metal helices in distillations where corrosive mixtures were being distilled. Glass helices are not manufactured in as many sizes as metal helices, and they are not as uniformly constructed. In packing columns with glass helices, care must be taken not to break them. More variation in the H.E.T.P. of glass helices will be noted owing in part to their fragility, which prohibits tamping to provide a uniformly packed section. Table II gives the operating characteristics of ⅛ in. glass helices.

TABLE II. Operating Characteristics of Glass Helices*

Column length, cm.	I.D., cm.	Throughput, ml./min.	Theor. plates	H.E.T.P., cm.	Holdup, ml./plate	Efficiency factor
43	1.0	2.7	11	3.9	0.70	230
43	1.0	7.8	11	3.9	0.77	610
80	1.2	3.7	21	3.8	—	—
80	1.2	11.3	18	4.3	—	—

*From A. L. Glasebrook and F. E. Williams in *Technique of Organic Chemistry*, Vol. 4, Interscience, New York, 1951.

Packing Supports

Adequate support for the packing is essential, but it must be so designed that the supporting device is not the limiting factor in the column throughout. The most elemental design is simply fusing four indentations in an enlarged section of the column as shown in Figure 5A.

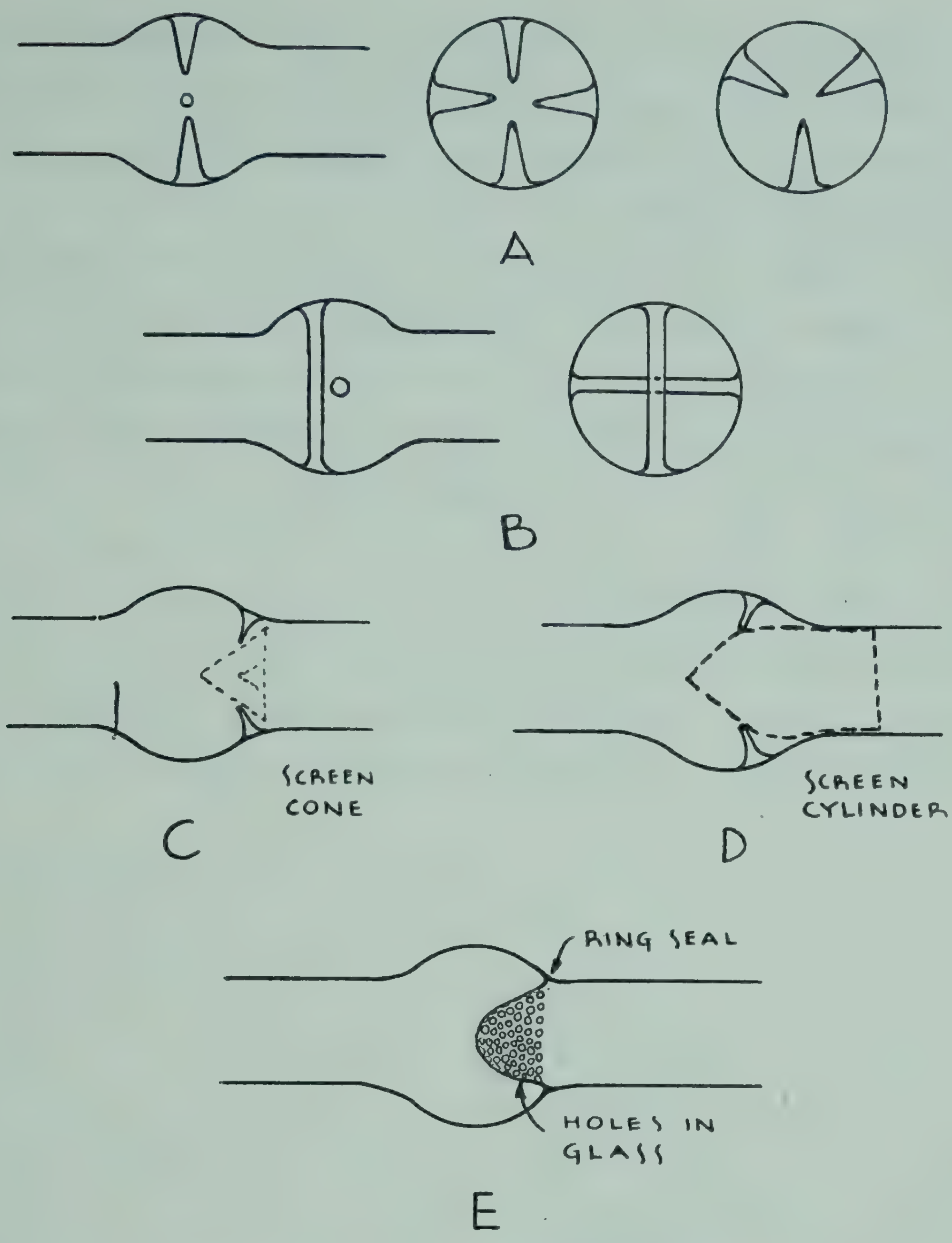

Figure 5. Column packing supports.

A few glass cylinders may be dropped into the column first to rest on these indentations and thus support the finer main packing. A similar device is to seal a glass cross in the enlarged section of column (Figure 5B). The rods of the cross do not need to touch each other. With these crossed rods a few glass cylinders are required to support the main packing.

When metal packings are used, a very satisfactory support is a corrosion-resistant cone of metal screen. The mesh of the cone must be smaller than the diameter of the packing to be used. In place of the cone, a cylinder of wire mesh may be used to support the packing, and either the cone or cylinder will provide a larger area for liquid and vapor flow than the cross-sectional area of the column, provided the column has been enlarged at the section where the cone or cylinder is placed. If a platinum cone is used to support the packing the column should be flushed with hydrogen sulfide to poison the platinum and eliminate the catalytic action of the platinum.

A glass cone can be sealed to the bottom of the column as shown in Figure 5E. The holes should be just small enough to support the packing and the sum of the area of the holes should be more than equal to the free space of the packed column, and preferably should be considerably larger.

Packing Glass Helices

Packing glass helices by hand becomes a tedious chore since it is necessary that each helix be free from entanglement with other helices. Figure 6 shows a convenient and easily made apparatus for packing helices. This was described by Allenby and L'Heureux.[12]

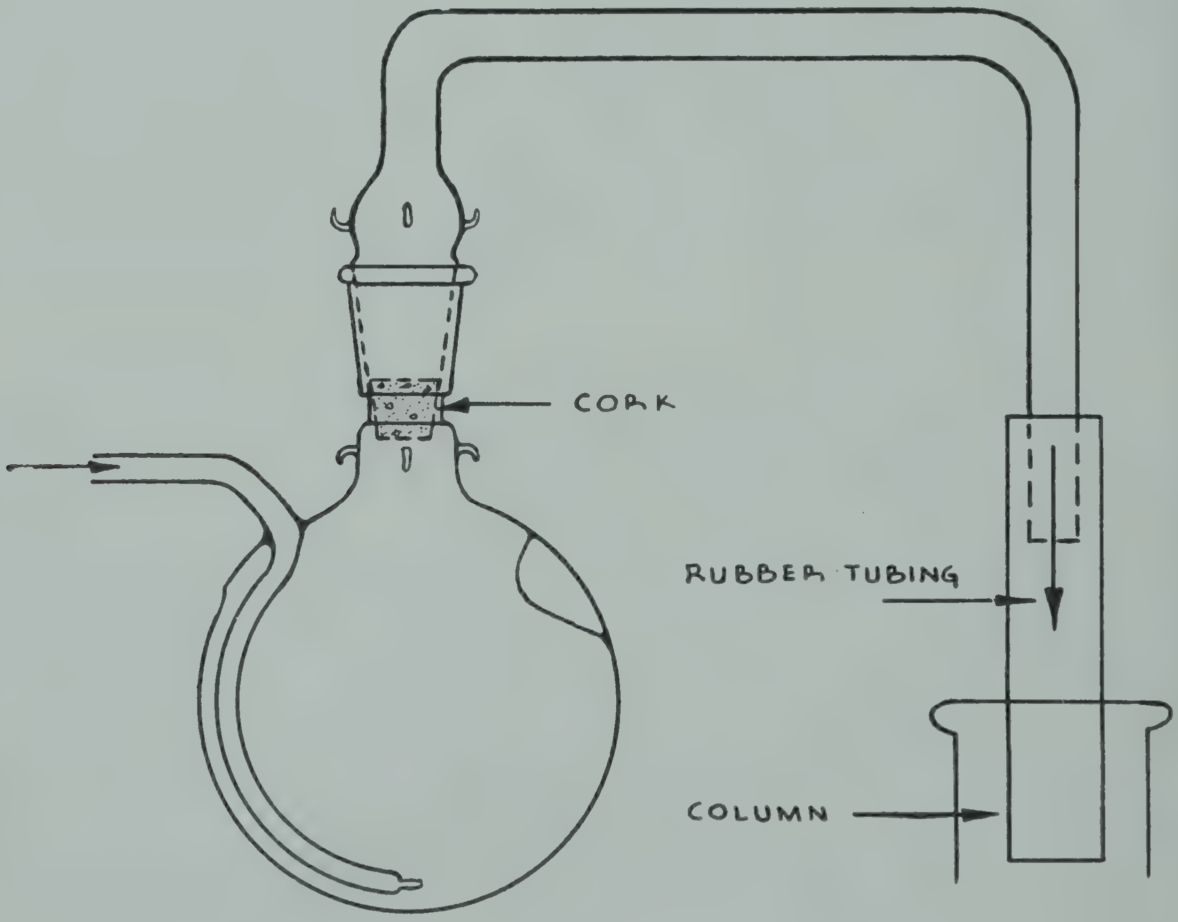

Figure 6. Apparatus for packing glass helices.

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[12] Allenby, O. C. W., and L'Heureux, C., *Anal. Chem.*, **22**, 1340 (1950).

Stedman Packing

Stedman[13] packing is made from wire gage which has been cut and stamped into truncated cones. A semicircular hole is cut in one side of the cone, extending about two-thirds of the distance from the edge of the cone to the plate center. The cones are assembled so the cut-out semicircular holes are located alternately on the opposite sides of the cell, forming a continuous vapor passageway. The cones are welded together and the efficiency of the packing depends in part on the accuracy with which this welding is done to form a straight series of cells. Precision-bore glass tubing is generally used for these columns to insure a closer fit between the packing and the containing tube. Stedman columns are sold by several scientific supply firms, among them Scientific Glass Apparatus Co.[14] and H. S. Martin Co.[15]

Chain Packing

Chain packing enjoys some use owing to its availability and the ease with which a column may be packed. According to Schicktanz[16] the efficiency of chain packing is increased by changing from a single-jack chain to a double-jack chain. An increase in the exposed effective surface and further increase in efficiency will be gained by changing to a jeweler's brass locket chain. However, McMillan[17] found that even the jeweler's chain was not as effective as a wire spiral packing. In addition, channeling and flooding of chain packing makes distillations more difficult to control.

Widmer Column[18]

A Widmer column consists of a glass spiral slipped into the annular space between two pieces of glass tubing. The hot vapors rise up through the outer jacket, pass down through a second jacket, and finally rise again through the column proper. See Figure 7.

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[13] Stedman, D. F., *Trans. Am. Inst. Chem. Engrs.*, **33**, 153 (1937).

[14] Bloomfield, New Jersey.

[15] Evanston, Illinois.

[16] Schicktanz, S. T., *J. Research, Natl. Bur. Standards* **11**, 89 (1933).

[17] McMillan, W. U., *J. Inst. Petroleum Technol.*, **22**, 616 (1936).

[18] Widmer, G., *Helv. Chim. Acta*, **7**, 59 (1924).

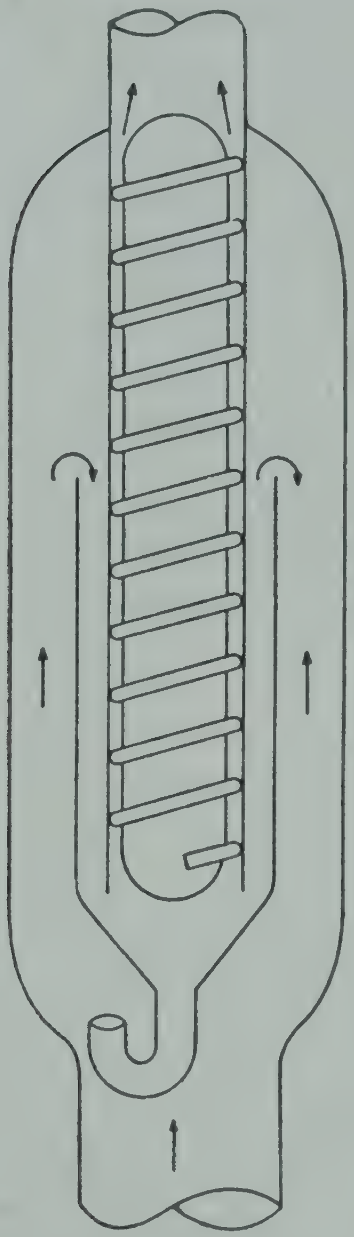

Figure 7. Widmer column. (From A. L. Glasebrook and F. E. Williams in *Technique of Organic Chemistry*, Vol. 4, Intersc., New York, 1951.)

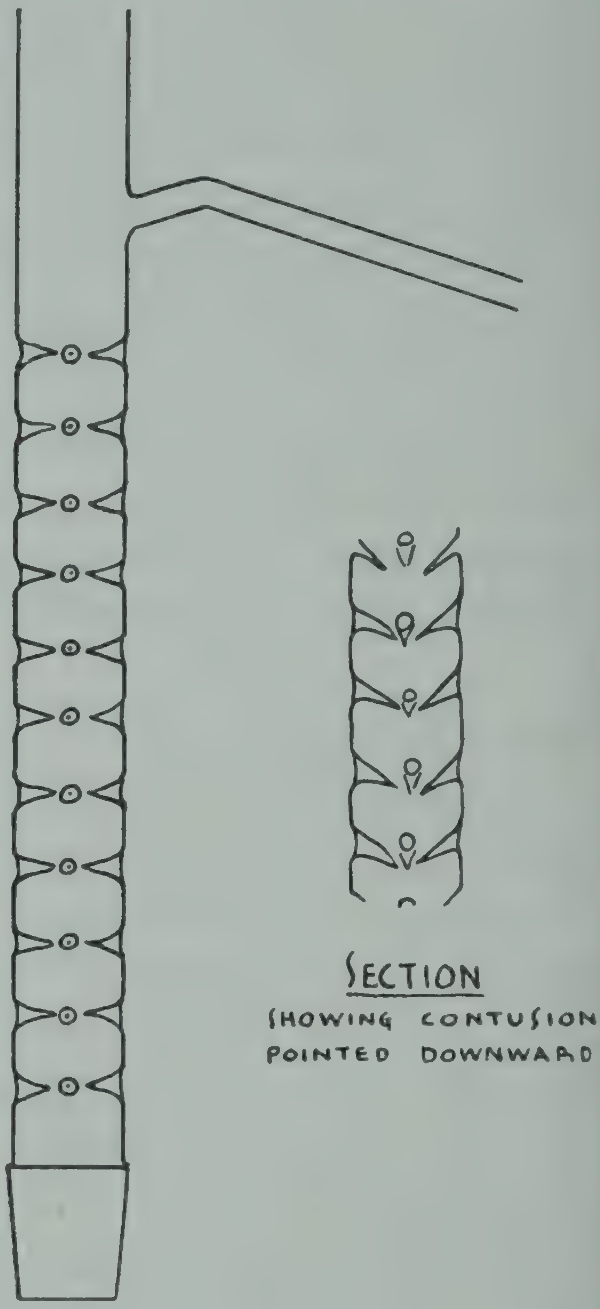

Figure 8. Vigreux column.

Vigreux Column

A Vigreux column is what is known as a distorted wall column. For ease of construction and operation, Vigreux columns are probably the most practical of all columns (see Figure 8). Vigreux columns are made by heating a spot on the glass tube and then pressing inward and slightly downward with a pointed carbon rod. The depth of the indentation should be less than half the diameter of the tube. A second indentation is made at an angle of 180° to the first. The tube is then rotated 90° and two more indentations pushed in, either slightly higher or slightly lower than the first two. This procedure is repeated until the rectifying section has been completely indented. Both Shrader and Ritzer[19], and Cooper and Fasce[20] have reported micro vacuum-jacketed Vigreux columns which gave good separation between materials boiling 10° C. apart. Fenske[21] and his co-workers distilled samples as small as 0.5 to 2.0 g. with a vacuum-jacketed column, and reported an H.E.T.P. of 13.7 cm. for a 1.1 cm. diameter Vigreux column.

However, Carney[22] recommends that if Vigreux columns are air-jacketed the jacket temperature should be held about 25° C. lower than reflux temperature. Because there is no packing the walls of the column serve as the surface area of contact, making it necessary for the walls to be maintained at such a temperature that appreciable quantities of material will condense on them, thus giving the ascending vapor opportunity to come in contact with the returning reflux.

Concentric-Tube Column

Concentric-tube columns are noted for their low holdup and high operating efficiency. In addition, their all-glass construction permits their being used for distilling corrosive materials. The high efficiency of these columns is obtained from the washing action between the vapor and liquid when they pass up and down in the narrow annular space between the two tubes. Under proper operating conditions, the liquid forms a thin film on the surface of both tubes, allowing intimate contact with the ascending vapors.

Concentric-tube columns were used as early as 1900 by

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[19] Shrader, S. A., and Ritzer, J. E., *Ind. Eng. Chem., Anal Ed.*, **11**, 54 (1939).

[20] Cooper, C. M., and Fasce, E. V., *Ind. Eng. Chem.*, **20**, 420 (1928).

[21] Fenske, M. R., Tongberg, C. O., and Quiggle, D., *Ind. Eng. Chem.* **26**, 1169 (1934).

[22] Carney, T. P., *Laboratory Fractional Distillation*, p. 63. Macmillan, New York, 1949.

Rosanoff[23] but they were not used to a large extent until 1937, when Craig[24] showed that the rectifying efficiency of these columns was excellent. In 1946 Naragon and Lewis[25] designed a concentric-tube column of high efficiency. The construction features of this column are shown in Figure 9.

Concentric-tube columns depend on the liquid vapor contact taking place in the small annular space between the two tubes; therefore, both tubes comprising the rectifying section must be uniform in diameter over their entire length in order to give a uniform annular space. It is also very important that the tubes be sealed in a vertical position in the column. During operation, the column must be mounted accurately to prevent the reflux from channeling in the column. Should channeling take place, the efficiency of the column is greatly reduced. A concentric-tube column is built by the Precision Distillation Apparatus Company.[26]

In this column the head is built into the vacuum jacket directly over the rectifying section, thus avoiding the possibility of the reflux's being condensed before reaching the cold finger (coil) condenser. It is designed primarily for total condensation, liquid take-off. A coil condenser directs the flow of condensate onto a glass plunger that is operated by a solenoid coil controlled from an electronic repeat timer. The metal bar energized by the solenoid coil is enclosed in glass; thus the distillate never comes in contact with metal. An equalizing line from the top of the column to the take-off line inside the vacuum jackets assures satisfactory operation of these columns at reduced pressures. As is true of all low-throughput columns, concentric-tube columns must be well insulated. When silvered vacuum jackets are used, satisfactory results may be obtained at temperatures up to

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[23] Rosanoff, M. A., Lamb, A. B., and Breithut, F. E., *J. Am. Chem. Soc.*, **31,** 448 (1909).

[24] Craig, L. C., *Ind. Eng. Chem., Anal Ed.*, **9**, 441 (1937).

[25] Naragon, E. A., Lewis, C. J., *Ind. Eng. Chem., Anal. Ed.*, **18,** 448 (1946).

[26] Precision Distillation Apparatus Co., 2304 Wilshire Blvd., Santa Monica, California.

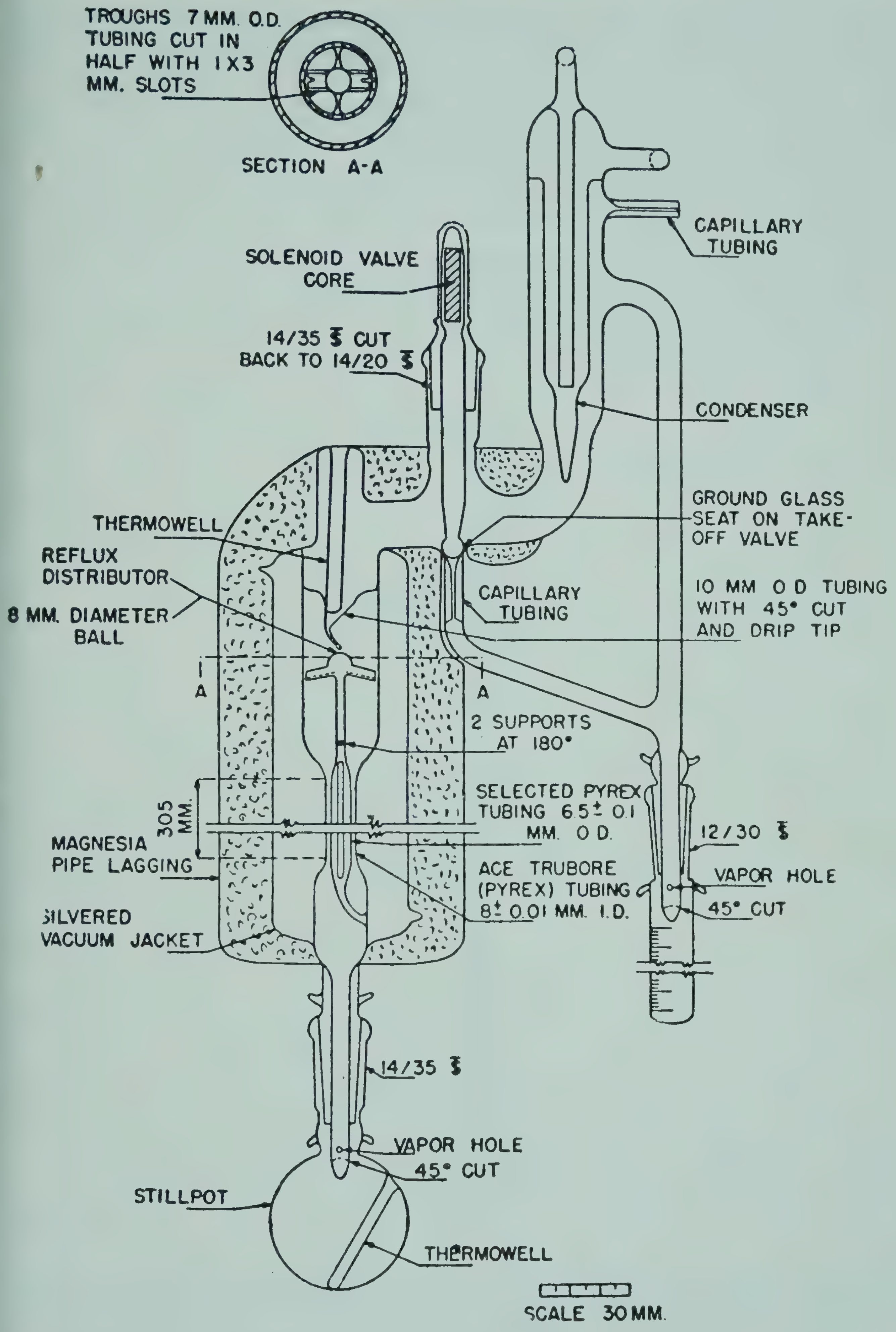
TROUGHS 7 MM. O.D. TUBING CUT IN HALF WITH 1 X 3 MM. SLOTS
SECTION A-A
SOLENOID VALVE CORE
14/35 § CUT BACK TO 14/20 §
CAPILLARY TUBING
CONDENSER
GROUND GLASS SEAT ON TAKE-OFF VALVE
THERMOWELL
REFLUX DISTRIBUTOR
8 MM. DIAMETER BALL
CAPILLARY TUBING
10 MM O D TUBING WITH 45° CUT AND DRIP TIP
A
A
2 SUPPORTS AT 180°
305 MM.
SELECTED PYREX TUBING 6.5 ± 0.1 MM. O D.
12/30 §
MAGNESIA PIPE LAGGING
ACE TRUBORE (PYREX) TUBING 8 ± 0.01 MM. I.D.
VAPOR HOLE
45° CUT
SILVERED VACUUM JACKET
14/35 §
VAPOR HOLE
45° CUT
STILLPOT
THERMOWELL
SCALE 30 MM.

Figure 9. Concentric-tube column. (From A. L. Glasebrook and F. E. Williams in *Technique of Organic Chemistry,* Vol. 4, Interscience, New York, 1951).

200° C. However, for reduced-pressure distillation and high temperatures (up to 400° C.), columns modified for use with electric heating mantles are recommended.

Because of the low operating holdup of the concentric tube columns, excellent micro columns may be made using this design. Figure 10 is a detailed drawing of the construction features of a micro concentric-tube column. This column was designed for distillations at atmospheric pressures only and has a theoretical plate efficiency of 43.0 plates as tested with a *n*-heptane-methylcyclohexane mixture.

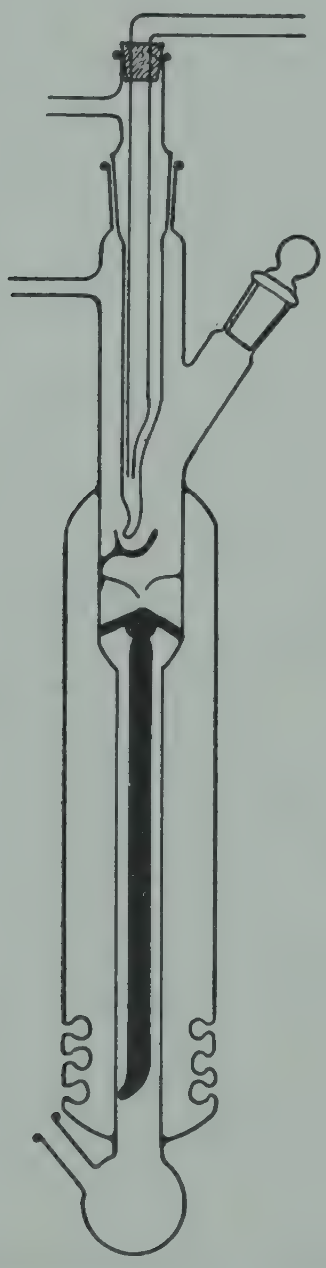

Figure 10. Micro concentric-tube column (Courtesy Precision Distillation Apparatus Co.).

Empty Tube Columns

The simplest type of rectifying column is that made from an empty tube. Rose[27] found that, in spite of the simplicity, such a column, if well insulated, might attain an efficiency as high as 30 plates per foot, provided the throughput was kept low. Owing to this low throughput, empty tube columns require the best insulation, and since they are extremely sensitive to any changes of the heat input to the kettle, a voltage regulator should be used in conjunction with a Variac for controlling the boiling pot heater. In addition, the kettle should be well insulated to protect it from drafts or temperature change in the room.

Oldershaw Perforated Plate Columns

In 1941 Oldershaw[28] described an all-glass perforated plate column (see Figure 11) with a reflux rate of high capacity. This column consists of a series of perforated plates (Figures 11A and 11B) having a baffle to direct the flow of liquid, a weir to maintain the level of liquid on each plate, and a drain pipe. Each plate has 42 holes, 0.85 mm. I.D., which are drilled by means of a hot tungsten wire—an operation which, when done by hand, is very time-consuming and tedious. Langdon and Tobin[29] reported on a method of molding perforated plates. These plates are made from Pyrex glass, ground and screened through a 360-mesh sieve. A special jig which provided the correct number of holes was made for forming the plates, and these blanks were baked at 925° C. for 5 to 6 minutes.

In the Oldershaw column the separate plates are carefully sealed in a tube so that each plate is level and the bead on the bottom of the drain pipe is resting on the plate below. In the Langdon column the plates are supported in the column by means of spacers and may be removed or added to at will.

Collins and Lantz,[30] in a series of tests to evaluate the Oldershaw perforated plate column in comparison with other

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[27] Rose, A., *Ind. Eng. Chem.*, **28**, 1210 (1936).

[28] Oldershaw, C. F., *Ind. Eng. Chem., Anal. Ed.*, **13**, 265 (1941).

[29] Langdon, W. M., and Tobin, D. J., *Ind. Eng. Chem., Anal. Ed.*, **17**, 801 (1945).

[30] Collins, F. C., and Lantz, V., *Ind. Eng. Chem., Anal. Ed.*, **18**, 673 (1946).

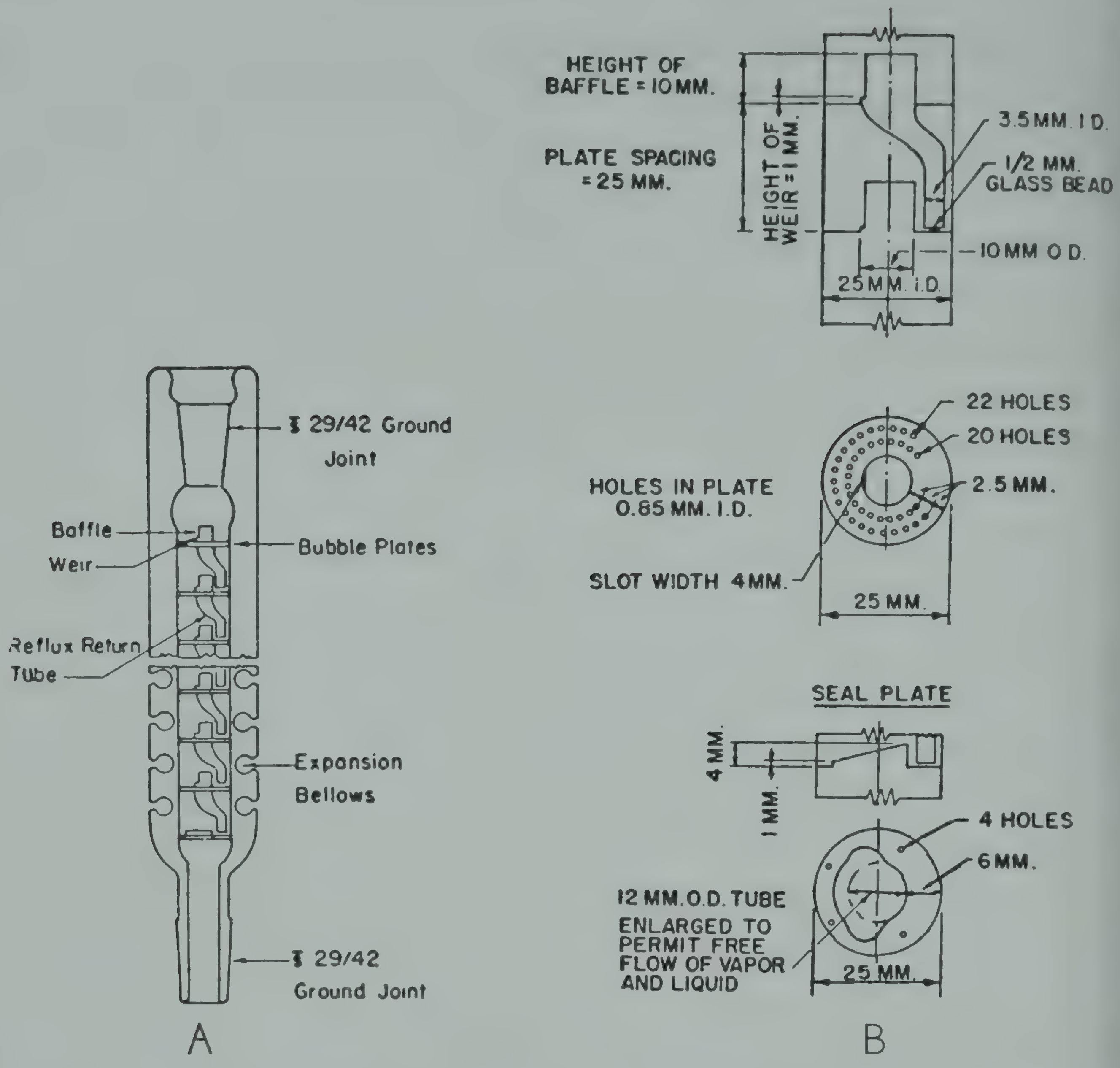
HEIGHT OF BAFFLE = 10 MM.
PLATE SPACING = 25 MM.
HEIGHT OF WEIR = 1 MM.
3.5 MM. I.D.
1/2 MM. GLASS BEAD
10 MM O.D.
25 MM. I.D.
22 HOLES
20 HOLES
2.5 MM.
HOLES IN PLATE 0.85 MM. I.D.
SLOT WIDTH 4 MM.
25 MM.
SEAL PLATE
4 MM.
1 MM.
4 HOLES
6 MM.
12 MM. O.D. TUBE ENLARGED TO PERMIT FREE FLOW OF VAPOR AND LIQUID
25 MM.
$ 29/42 Ground Joint
Baffle
Weir
Bubble Plates
Reflux Return Tube
Expansion Bellows
$ 29/42 Ground Joint
A
B

Figure 11. Oldershaw perforated plate columns. (From A. L. Glasebrook and F. E. Williams in *Technique of Organic Chemistry*, Vol. 4, Interscience, New York, 1951.)

types of columns used for analytical distillation, found that the Oldershaw column had an exceptionally high throughput capacity and a low operating holdup per theoretical plate. They also noted that the efficiency of an Oldershaw column is nearly independent of the throughput rate and shows no significant variation from column to column. This is especially important in laboratories where reproducibility of results is of primary importance. From the studies of Collins and Lantz an improved Oldershaw column was developed having 82 holes 0.89 mm. I.D. in place of the original 42 holes per plate as described by Oldershaw.

Instructions for the construction of the Oldershaw perforated columns are included in the original literature. These

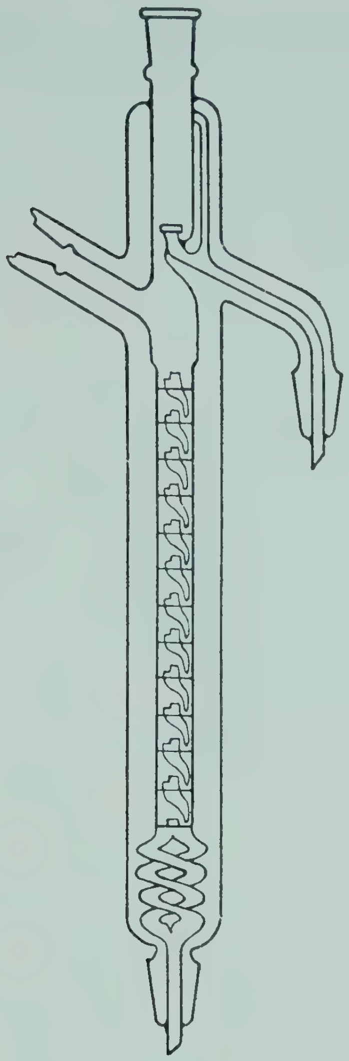

Figure 11. (Continued.)

columns may be made by an experienced glassblower; they are, however, produced commercially. Figure 11C shows a vacuum-jacketed column with a built-in electronically controlled head as manufactured by the Precision Distillation Apparatus Co.[26]

VARIABLES AFFECTING OPERATING CHARACTERISTICS

A number of variables affecting the operating characteristics of a column should be taken into consideration before designing any new equipment.

Diameter

Increasing the diameter of a column increases the throughput. Ward[31] states, "The capacities of columns are proportional to the square of their diameters. The holdup, however, increases in somewhat the same ratio, and thus there is some loss in fractionating efficiency." An increase in the diameter of a column decreases the vapor velocity for the same throughput.

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[31] Ward, C. C., *U. S. Bur. Mines Techn. Paper,* **600**, 1939.

Column Height

An increase in the height of a column increases the number of theoretical plates obtained from the column. However, such an increase follows the law of diminishing returns, i.e., each additional section of length is less efficient than the preceding section. The increase in H.E.T.P. is smaller, however, than the gain in efficiency owing to the increased length. Thus a long column, when conditions permit the increased holdup, is preferable to a short column, since at total reflux the degree of separation is increased logarithmically, not linearly, with the number of perfect plates. Because the number of perfect plates is a function of its length, the best results are obtained with as long a column as possible, taking into account the amount of material to be distilled as well as construction facilities.

Holdup

The amount of holdup in any column is important, since this measures the sharpness of separation obtained. Carney[32] states, "the maximum allowable holdup should not exceed, by volume, 10 % of either of the components being separated." The volume of the intermediate cut between the pure components will be equal to the volume of holdup. When small batches of material are being distilled, a large intermediate cut is a matter of concern.

Pressure Drop

At atmospheric pressures a slight difference in pressures between the head and the still pot is of small concern. However, an increase in the pressure drop does increase the pressure in the still pot, which, in turn, increases the temperature of the distillation. Chilton and Colburn[33] estimated that only 10 % of the pressure drop in a packed column is due to frictional resistance, the rest being due to contraction and expansion losses in the vapor flowing through the irregular free spaces formed by the packing. Glasgow and Schicktanz[34] used the pressure drop from head to still pot to control the heat input to the still pot, thus controlling the throughput.

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[32] Carney, T. P., *Laboratory Fractional Distillation*, p. 96. Macmillan, 1949.

Vapor Velocity

An increased vapor velocity or throughput results in less efficiency of separation and a higher H.E.T.P. The loss of efficiency could be attributed to inefficient contact between the liquid and the vapor, caused either by channeling or by the fact that the vapor velocity is so great that the time of contact necessary for efficient heat exchange is too short. This loss of efficiency with increased vapor velocity points out the need for accurate control of heat input and boil-up rate with many columns. This is especially true of high-efficiency columns being operated to give the maximum possible separations.

Distillation Rate

The distillation rate is a function of the vapor velocity and the reflux ratio. With a high vapor velocity, the distillation rate increases but the time of contact between the vapor and liquid is decreased. A high reflux ratio increases the length of time necessary for a given distillation, but it also increases the degree of fractionation.

Water in Material To Be Distilled

Small amounts of water in organic liquids should be thoroughly removed before attempting a precise fractionation. Any water not removed will steam distill with the first part of the distillation and give erroneous still head temperature readings. In many cases water will separate from the organic distillate and cling tenaciously to the walls of the still head, hindering smooth operation. Frequently the separated water will redistill over and over again in the head before finally passing into the take-off condenser, where it may again cling to the walls.

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[33] Chilton, T. H., and Colburn, A. P., *Ind. Eng. Chem.*, **23**, 917 (1931).

[34] Glasgow, A. R. Jr., and Schicktanz, S. T., *J. Research Natl. Bur. Standards*, **14**, 393 (1937).

Preflooding the Column at the Start

Preflooding consists of superheating the still pot to the extent that the ascending vapors prevent the reflux from returning to the still pot, resulting in liquid's completely filling the column and thoroughly wetting the packing with the distillant—a condition which is necessary for the maximum operating efficiency of the packing.

For random-packed columns, micro columns, and especially the thin-film concentric-tube columns, preflooding should be done before each distillation to obtain the maximum plate efficiency from the packing and the column. Bubble plate and Oldershaw type perforated plate columns are not improved by preflooding.

Preflooding is accomplished by setting the heat to the still pot high and bringing the sample to a boil in the shortest time possible. As soon as boiling begins, or when the boiling point is nearly reached as shown by a thermometer in the boiling flask, the voltage used in heating the flask should be reduced to about twice the voltage expected to be used during the distillation. Actual flooding of the column should not be too rapid, so that liquid will not be carried over into the receiver and trap; flooding, however, should be continued until the liquid completely fills the column and head up to the condensing section. At this point heat on the still pot should be reduced to allow the column to drain, and as the liquid returns to the still pot it thoroughly wets the packing. Heat on the still pot should be set at normal as soon as the excessive drainage of liquid stops and at no time should refluxing be allowed to stop completely; otherwise the favorable results obtained by the preflooding will be partially or completely lost.

For some densely packed columns a second preliminary preflooding is recommended before equilibrating the column. The procedure for a second preflooding is the same as for the first and starts as soon as the reflux has returned to normal following the first flooding.

EVALUATION OF FRACTIONATING COLUMNS

A satisfactory test for evaluating a fractionating column is to determine the number of theoretically perfect plates necessary to make the same separation as the column, assuming that operating conditions are identical. To make this determination, a binary mixture is distilled until equilibrium has been reached. By withdrawing and analyzing samples of the distillate and of the liquid from the still pot, the equivalent number of theoretical plates necessary to make this separation can be calculated, by means of the equation shown later in this section.

The number of theoretical plates in a column indicates the separation that can be made with that particular column. However, this does not give a true comparison between columns of different lengths. Tests with two columns of different lengths may show that the longer column has more theoretical plates and thus will give a better overall separation, but that the shorter column gives a better separation per unit length. For this reason, comparison of columns is usually based on the Height Equivalent to a Theoretical Plate (H.E.T.P.). This is the height required to make a separation equivalent to that effected by a fractionating column of "one-plate" rating. Mathematically, the H.E.T.P. is the length of the column (either in inches or centimeters) divided by the numerical theoretical-plate value of that section.

The H.E.T.P. is also useful in rating the efficiency of columns according to the degree of separation, but, unfortunately, it is affected by the rate of distillation. This makes rating of various types of columns difficult unless one standard operating procedure is used. One method especially adapted to high-throughput, random-packed columns is to run each column at total reflux and at a point just below flood point. Values obtained under these conditions are very conservative, and better efficiency will be obtained at lower throughput rates. However, reasonably accurate comparisons between columns can be made in this way. With low-holdup, slow-throughput columns, as exemplified by the concentric-tube columns, the results obtained by operating at just below the flood point will be so low as to be worthless.

Columns may be tested at either total or partial reflux. In most cases it is advantageous to rate a column under total reflux, since results obtained are comparable and may be used for comparisons between different columns. A column tested under total reflux will show a higher degree of efficiency than the same column under partial reflux. It is safe to assume, however, that the column with the higher degree of efficiency under total reflux will also be more efficient under actual operating conditions. To determine in an individual column what separation may be expected under operating conditions, it is best to test the column with the reflux ratio and boil-up rate to be used during the actual distillation.

CHOICE OF A BINARY TEST MIXTURE

Any test mixture chosen for evaluating columns should meet the following conditions: it should be easily obtained in the pure state, if possible, or it should be easily purified; and the vapor-liquid equilibrium data should be available in well-established literature.

Carbon Tetrachloride and Benzene

Carbon tetrachloride (b.p. 76.7° C.) and benzene (b.p. 80.1° C.) is a satisfactory test mixture. Both components can be obtained pure enough to use without preliminary distillation and, in addition, both components are relatively cheap. A composition of 91.8 mole per cent of carbon tetrachloride must be avoided, however, since this composition forms a constant boiling mixture (azeotrope) which cannot be separated. One other disadvantag of carbon tetrachloride is that it will react with aluminum packing and will corrode some other metal packings if used over a long period of time. The carbon tetrachloride-benzene mixture is probably the most widely used of all test mixtures and is suitable for columns up to about 25 theoretical plates. Analysis by both refractive index and specific gravity is easy and accurate.

TABLE III. Carbon Tetrachloride-Benzene Liquid Vapor Data*

Vapor Comp.	Liquid Comp.	n_D^{25}
0.000	0.000	1.49794
.122	.100	1.49392
.233	.200	1.48997
.337	.300	1.48589
.437	.400	1.48178
.534	.500	1.47772
.628	.600	1.47369
.720	.700	1.46963
.812	.800	1.46555
.831	.820	—
.850	.840	—
.869	.860	—
.888	.880	—
.907	.900	1.46142
.918	.918	—
1.000	1.000	1.45732

This mixture has probably been more widely used for testing than any other. It is suitable for columns with up to about 25 plates. Analysis is almost always by refractive index, but density may be used. Initial composition should be such that still composition at the time of taking samples is not much less than 6 mole per cent carbon tetrachloride, and the condenser sample should not have more than about 80 mole per cent carbon tetrachloride. At these concentrations one theoretical plate corresponds to about 0.0005 change in refractive index. The azeotropic composition of 91.8 mole per cent carbon tetrachloride must be entirely avoided. Serious corrosion of aluminum or iron packings or columns is likely to occur with any concentration of this test mixture.

*From A. and E. Rose in *Technique of Organic Chemistry,* Vol. 4, Interscience, New York, 1951.

Table III gives the data for carbon tetrachloride-benzene test mixture. (All compositions are in mole fraction of carbon tetrachloride.)

TABLE IV. *n*-Heptane-Methylcyclohexane Liquid-Vapor Data*

Vapor-liquid equilibrium data		Data for analysis of samples		
Vapor compn.	Liquid compn.	n_D^{20}	d_4^{20}	B.p., °C. (760 mm.)
	0.0000	1.4232	0.769	100.80
0.0350	.0310			
.0620	.0580			
	.0787	1.4200	.7613	100.55
.1030	.0950			
.1430	.1330			
	.1638	1.4165	.7535	100.35
.1920	.1800			
.2290	.2260			
	.2486	1.4135	.7454	100.15
.2890	.2715			
.3330	.3170			
	.3372	1.4100	.7377	—
.3810	.3630			
.4200	.4010			
	.4126	1.4075	.7312	99.70
.4750	.4560			
.5210	.5010			
	.5186	1.4036	.7216	99.2ä
.5780	.559ä			
.6180	.5990			
	.6056	1.4004	.7145	99.00
.6660	.6470			
	.6993	1.3970	.7069	98.85
.7280	.7090			
.7710	.7560			
	.7942	1.394?	.6992	98.60
.8100	.7960			
.8535	.8430			
.8900	.8790			
.9130	.9060			
.9400	.9310			
	.9338	1.3899	.6884	98.50
.9625	.9540			
.9860	.9800			
	1.0000	1.3878	.6839	98.40

This mixture is useful for testing columns with about 10 to 90 plates. It is particularly suitable for columns distilling close-boiling hydrocarbons. Analysis is by refractive index or density.

*From A. and E. Rose in *Technique of Organic Chemistry*, Vol. 4, Interscience, New York, 1951.

n-Heptane-methylcyclohexane

This mixture is used for columns having a larger number of theoretical plates than can be calculated using the benzene-carbon tetrachloride mixture (i.e., 10 to 90 theoretical plates). Table IV gives the liquid-vapor equilibria data for this mixture.

METHODS FOR DETERMINING THEORETICAL PLATES

Graphic Method

McCabe and Thiele[35] published data for the graphic determination of the number of theoretical plates in a column. Figure

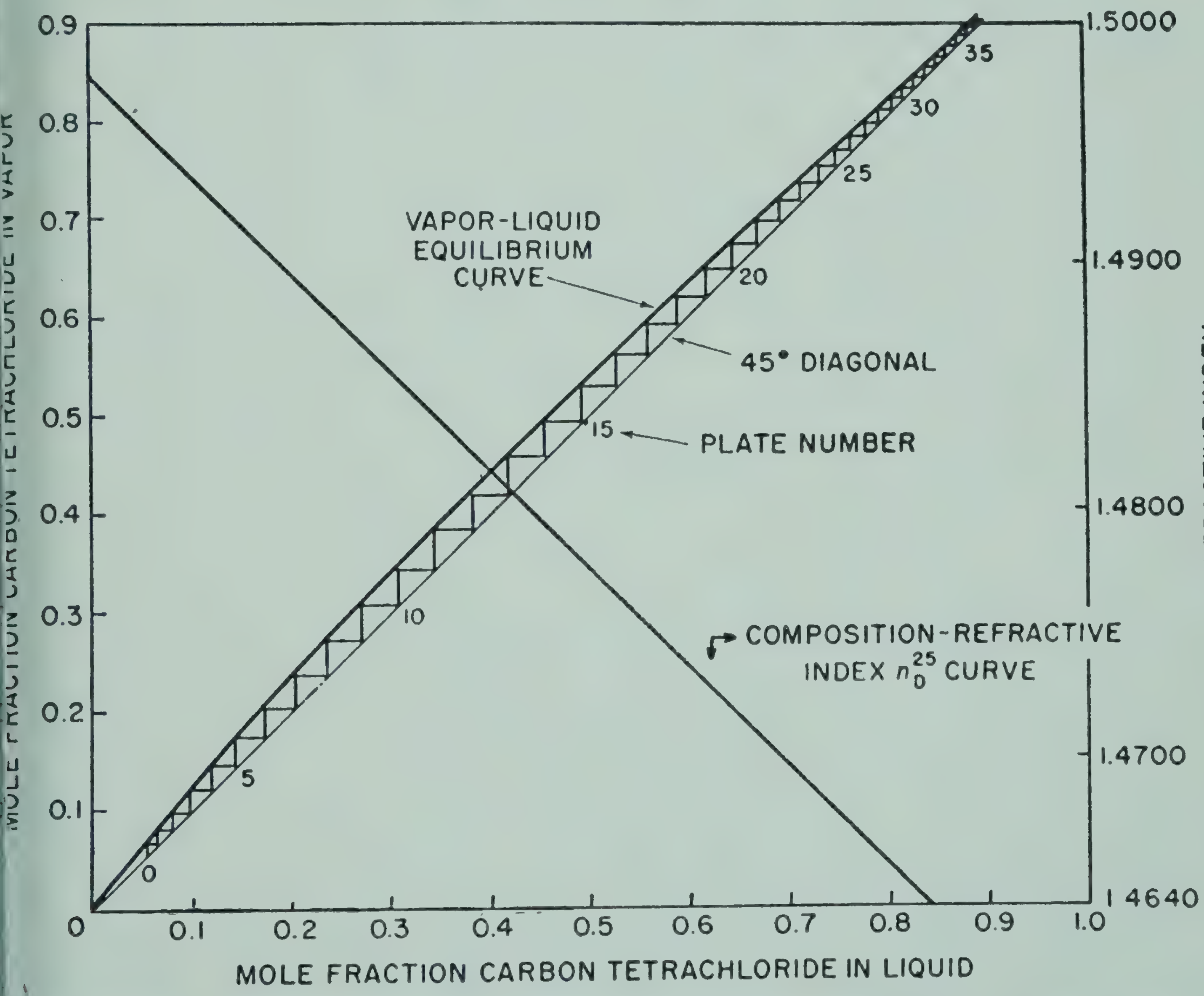

Figure 12. Mole fraction of carbon tetrachloride in liquid. (From A. and E. Rose in *Technique of Organic Chemistry*, Vol. 4, Interscience, New York, 1951).

[35] McCabe, W. L., and Thiele, E. W., *Ind. Eng. Chem.*, **17**, 605 (1925).

TABLE V. Benzene-Carbon Tetrachloride Index versus Plate Number *

n_D^{25}	00	01	02	03	04	05	06	07	08	09
	Plate number									
1.4600										
10						37.7	37.4	37.1	36.8	36.6
20	36.3	36.1	35.8	35.6	35.4	35.2	35.0	34.8	34.5	34.3
30	34.0	33.8	33.6	33.3	33.1	32.8	32.6	32.4	32.1	31.9
40	31.7	31.5	31.3	31.1	30.9	30.6	30.4	30.2	29.9	29.7
50	29.5	29.3	29.1	28.9	28.7	28.5	28.3	28.1	27.9	27.7
60	27.5	27.3	27.1	27.0	26.8	26.6	26.5	26.3	26.2	26.1
70	25.9	25.7	25.5	25.4	25.2	25.0	24.9	24.7	24.6	24.5
80	24.3	24.2	24.0	23.9	23.8	23.7	23.5	23.4	23.3	23.2
90	23.1	23.0	22.9	22.7	22.6	22.5	22.4	22.3	22.2	22.0
1.4700	21.9	21.8	21.7	21.6	21.5	21.4	21.3	21.2	21.0	20.9
10	20.8	20.7	20.6	20.5	20.5	20.3	20.2	20.1	20.0	19.9
20	19.8	19.8	19.7	19.7	19.6	19.5	19.4	19.3	19.2	19.1
30	19.0	18.9	18.8	18.8	18.7	18.6	18.5	18.4	18.3	18.2
40	18.1	18.0	17.9	17.8	17.7	17.7	17.6	17.5	17.4	17.3
50	17.3	17.2	17.1	17.0	16.9	16.9	16.8	16.7	16.7	16.6
60	16.5	16.5	16.4	16.3	16.2	16.2	16.1	16.0	15.9	15.9
70	15.8	15.8	15.7	15.6	15.5	15.5	15.4	15.3	15.2	15.1
80	15.1	15.0	14.9	14.9	14.8	14.7	14.6	14.5	14.5	14.4
90	14.4	14.3	14.2	14.1	14.0	14.0	13.9	13.8	13.8	13.7
1.4800	13.7	13.6	13.6	13.5	13.4	13.4	13.3	13.2	13.1	13.1
10	13.0	12.9	12.9	12.8	12.8	12.7	12.7	12.6	12.5	12.4
20	12.4	12.3	12.2	12.2	12.1	12.0	12.0	11.9	11.9	11.8
30	11.7	11.7	11.6	11.6	11.5	11.4	11.4	11.3	11.2	11.1
40	11.0	11.0	10.9	10.9	10.8	10.8	10.7	10.6	10.6	10.5
50	10.4	10.3	10.3	10.2	10.1	10.0	10.0	9.9	9.9	9.8
60	9.8	9.7	9.6	9.5	9.5	9.4	9.3	9.3	9.2	9.1
70	9.1	9.0	8.9	8.9	8.8	8.8	8.7	8.6	8.5	8.5
80	8.4	8.3	8.2	8.2	8.1	8.0	8.0	7.9	7.8	7.8
90	7.7	7.7	7.6	7.5	7.4	7.3	7.2	7.1	7.1	7.0
1.4900	6.9	6.9	6.8	6.8	6.7	6.6	6.5	6.4	6.3	6.2
10	6.1	6.1	6.0	5.9	5.8	5.7	5.6	5.5	5.4	5.3
20	5.2	5.1	5.0	4.9	4.8	4.7	4.6	4.5	4.4	4.3
30	4.2	4.1	4.0	3.9	3.8	3.7	3.6	3.5	3.3	3.2
40	3.1	2.9	2.8	2.7	2.5	2.4	2.3	2.2	2.0	1.9
50	1.9	1.6	1.4	1.2	1.0	0.9	0.7	0.5	0.3	0.2
60										

*From A. and E. Rose in *Technique of Organic Chemistry*, Vol. 4, Interscience, New York, 1951.

12 gives the vapor-liquid diagram for a test mixture of benzene and carbon tetrachloride, showing the theoretical plates stepped off from the composition 0.05 to 0.90 mole fraction of carbon tetrachloride. These steps are numbered, from 0 for the lowest value to 35 for the highest. This graph is read by marking off the step corresponding to the composition of the liquid from the kettle and the composition of the distillate, then subtracting the smaller plate number from the larger. Thus, if the refractive indices of the samples from the column are 1.4700 and 1.4900, the corresponding plate numbers are 21.9 and 6.9, giving 15.0 plates. When the column being tested has a total condenser, one theoretical plate is subtracted for the still. When a partial condenser is used, two plates should be subtracted, one for the still and one for the condenser.

Tabular Method

The use of a graph is entirely eliminated by the use of the tabular method described by Rose and Rose.[36] The refractive indices are located on Table V and the steps are shown directly. Refractive indices 1.4700 and 1.4900 give steps 21.9 and 6.9 as before. Values are given only to the nearest 0.1 plate. However, this is as precise as is necessary because of the variations in the H.E.T.P. measurements.

Algebraic Method

Columns with 30 plates or more should be calculated by means of the algebraic method of calculating plate efficiency, and the equation proposed by Fenske[37] gives satisfactory results. This equation, however, can be used only if the test mixture is ideal, that is, if it obeys Raoult's law, which states that the partial pressure of any component is equal to the product of the vapor pressure of the pure component and its mole fraction in solution. A test mixture of methylcyclohexane and *n*-heptane meets these conditions, and this mixture may be used satisfactorily in testing columns ranging from 10 to 90 theoretical plates.

[36] Rose, A., and Rose, E., in *Technique of Organic Chemistry*, Vol. **4**, p. 73. Interscience, New York, 1951.

[37] Fenske, M. R., *Ind. Eng. Chem.*, **24**, 482 (1932).

TABLE VI. Refractive Index versus Per Cent *n*-Heptane in Liquid

1.4232–0.0
1.4230–0.5
1.4228–1.0
1.4226–1.5
1.4224–2.0
1.4222–2.5
1.4220–3.0
1.4218–3.5
1.4216–4.0
1.4214–4.5
1.4212–5.0
1.4210–5.5
1.4208–6.0
1.4206–6.5
1.4204–7.0
1.4202–7.5
1.4200–8.0
1.4198–8.5
1.4196–9.0
1.4194–9.5
1.4192–10.0
1.4190–10.5
1.4188–11.0
1.4186–11.5
1.4184–12.0
1.4182–12.5
1.4180–13.0
1.4178–13.5
1.4176–14.0
1.4174–14.5
1.4172–15.0
1.4170–15.5
1.4168–16.0
1.4166–16.5
1.4164–17.0
1.4162–17.5
1.4160–18.0
1.4158–18.5
1.4156–19.0
1.4154–19.5
1.4152–20.0
1.4151–20.5
1.4149–21.0
1.4148–21.5
1.4146–22.0
1.4144–22.5
1.4142–23.0
1.4140–23.5
1.4138–24.0
1.4136–24.5
1.4135–25.0
1.4133–25.5
1.4131–26.0
1.4129–26.5
1.4128–27.0
1.4126–27.5
1.4124–28.0
1.4122–28.5
1.4120–29.0
1.4119–29.5
1.4117–30.0
1.4115–30.5
1.4113–31.0
1.4111–31.5
1.4109–32.0
1.4107–32.5
1.4106–33.0
1.4104–33.5
1.4102–34.0
1.4100–34.5
1.4098–35.0
1.4096–35.5
1.4094–36.0
1.4092–36.5
1.4091–37.0
1.4089–37.5
1.4087–38.0
1.4085–38.5
1.4083–39.0
1.4081–39.5
1.4079–40.0
1.4078–40.5
1.4076–41.0
1.4074–41.5
1.4072–42.0
1.4071–42.5
1.4069–43.0
1.4067–43.5
1.4065–44.0
1.4063–44.5
1.4061–45.0
1.4059–45.5
1.4058–46.0
1.4056–46.5
1.4054–47.0
1.4052–47.5
1.4050–48.0
1.4049–48.5
1.4047–49.0
1.4045–49.5
1.4043–50.0
1.4041–50.5
1.4039–51.0
1.4037–51.5
1.4036–52.0
1.4034–52.5
1.4032–53.0
1.4030–53.5
1.4028–54.0
1.4026–54.5
1.4024–55.0
1.4022–55.5
1.4020–56.0
1.4019–56.5
1.4017–57.0
1.4015–57.5
1.4013–58.0
1.4011–58.5
1.4009–59.0
1.4007–59.5
1.4006–60.0
1.4004–60.5
1.4002–61.0
1.4000–61.5
1.3998–62.0
1.3996–62.5
1.3995–63.0
1.3993–63.5
1.3991–64.0
1.3989–64.5
1.3987–65.0
1.3985–65.5
1.3983–66.0
1.3982–66.5
1.3980–67.0
1.3978–67.5
1.3976–68.0
1.3974–68.5
1.3972–69.0
1.3970–69.5
1.3969–70.0
1.3968–70.5
1.3966–71.0
1.3965–71.5
1.3963–72.0
1.3961–72.5
1.3960–73.0
1.3959–73.5
1.3957–74.0
1.3955–74.5
1.3954–75.0
1.3952–75.5
1.3951–76.0
1.3949–76.5
1.3948–77.0
1.3946–77.5
1.3945–78.0
1.3944–78.5
1.3942–79.0
1.3941–79.5
1.3939–80.0
1.3938–80.5
1.3936–81.0
1.3935–81.5

TABLE VI. (Continued)

1.3933–82.0	1.3919–86.5	1.3906–91.0	1.3892–95.5
1.3932–82.5	1.3918–87.0	1.3905–91.5	1.3890–96.0
1.3930–83.0	1.3916–87.5	1.3903–92.0	1.3889–96.5
1.3929–83.5	1.3915–88.0	1.3901–92.5	1.3887–97.0
1.3927–84.0	1.3914–88.5	1.3900–93.0	1.3885–97.5
1.3926–84.5	1.3912–89.0	1.3899–93.5	1.3884–98.0
1.3924–85.0	1.3911–89.5	1.3897–94.0	1.3882–98.5
1.3923–85.5	1.3909–90.0	1.3895–94.5	1.3880–99.0
1.3921–86.0	1.3908–90.5	1.3893–95.0	1.3879–99.5
			1.3877–100.0

From the data given in Table IV (methylcyclohexane–*n*-heptane liquid-vapor data), a diagram showing theoretical plates at total reflux was made, and from this diagram in turn was made Table VI to simplify establishing the percentage of *n*-heptane in each sample taken from the kettle and in the distillate. To determine theoretical plates by the algebraic method proposed by Fenske, the following terms are used:

XF^1 = mole fraction of *n*-heptane in the still pot.
XF^2 = mole fraction of methylcyclohexane in the still pot.
XC^1 = mole fraction of *n*-heptane in the distillate.
XC^2 = mole fraction of methylcyclohexane in the distillate.
(α) = 1.07 = relative volatility of *n*-heptane and methylcyclohexane (ratio of their vapor pressures)—value determined by Beatty and Calingaert.
$N+1$ = total number of theoretical plates, including the column.
N = number of theoretical plates in the column.

The Fenske equations is:

$$\frac{XC^1}{XC^2} = (\alpha)^{N+1}\frac{XF^1}{XF^2}.$$

A Procedure for Determining the Number of Theoretical Plates.

A mixture of *n*-heptane and methylcyclohexane is refluxed in the column until equilibrium has been established. Since the time required for any column to equilibrate is a function of the holdup, the throughput, and the difficulty of the separation being attempted, no general rule can be advanced as to the time necessary for equilibrium to be attained. The time of this reflux period will vary between columns and with the same column under different operating conditions. The approach to equilibrium can be followed by observing the change and final stability of the composition in very small samples taken from the head of the column. Coulson[38] developed a rough method of calculating the time required for a column to come to equilibrium.

When equilibrium has been attained, approximately 1 ml. of liquid is withdrawn from both the head and still pot as nearly simultaneously as possible. The sample of distillate should be withdrawn slowly, so that equilibrium is maintained, while the sample from the still pot is withdrawn from a tube extending below the surface of the liquid (see Figure 13). Both lines should have been previously cleared of stagnant liquid. Analysis of each sample by refractive index will give the percentage of *n*-heptane when checked against Table VI. For example, if the analysis of the samples by refractive index showed a refractive index of the distillate to be 1.3948 and that of the still pot liquid to be 1.4024, Table VI shows these values to correspond to 77.0 and 55.0 mole

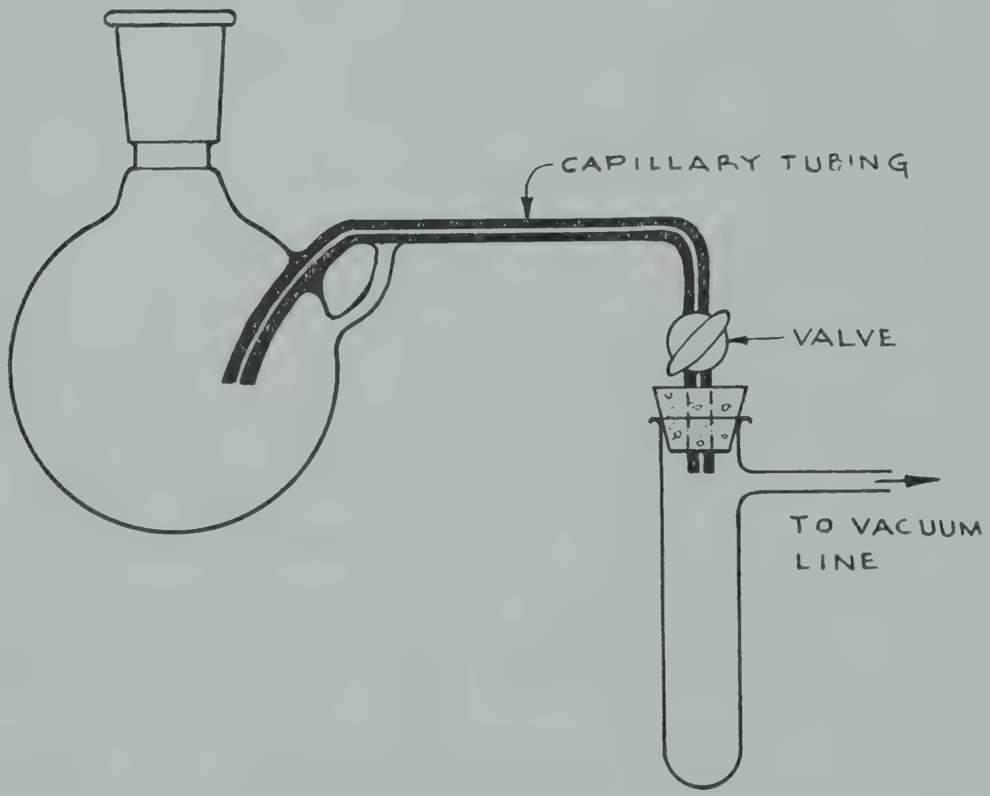

Figure 13. Still pot for the removal of samples.

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[38] Coulson, A. E., *J. Soc. Chem. Ind.* (*London*), **64**, 101 (1945).

fraction of *n*-heptane in the distillate and still pot, respectively. Thus according to the Fenske equation:

	Refractive Index	Mole Fraction of Component
Distillate	1.3948	77
Still pot	1.4024	55

$$\frac{55}{45} \times (1.07)^{n+1} = \frac{77}{23} \qquad \text{or} \qquad 1.23 \times 1.07^{n+1} = 3.35.$$

$$1.07^{n+1} = \frac{3.35}{1.23}; \qquad \text{thus } 1.07^{n+1} = 2.73.$$

$$(n+1) \times (\log 1.07) = \log 2.73;$$

$$n+1 = \frac{0.43616}{0.03019}; \qquad n+1 = 14.5.$$

After one theoretical plate for the column is subtracted, the answer is 13.5 theoretical plates.

DISTILLATION UNDER REDUCED PRESSURE

For ordinary distillations the preferred pressure is of course atmospheric, provided it can be maintained without the sample's decomposing or without the distillation's having to be carried out at excessively high temperatures. The principal advantage of distilling under reduced pressure is in the lower boiling point for the material being distilled. Because the pressure of the vapor varies logarithmically as the inverse of its absolute temperature, the lower the pressure at which the distillation is carried out, the greater the decrease in boiling temperature. Other advantages of reduced-pressure distillation is the lessened danger that material will oxidize or decompose during the distillation.

Disadvantages

The throughput or capacity of any column is considerably less at reduced pressure than at atmospheric pressure; consequently, more time is required to fractionate at a desired reflux rate. The sharpness of a separation in any given distillation depends on the same factors regardless of the pressure used. Usually under reduced pressure the transition between pure component plateaus is not as sharp and as easy to follow as in atmospheric distillations.

Choice of Columns

According to Feldman, Myles, Wender, and Orchin[39] most of the conventional types of columns and column packings are useful down to pressures of a few centimeters of mercury. Below this none give satisfactory performance, and specially designed vacuum columns should be used. Concentric-tube columns have been used successfully for separating materials boiling from 180° to 250° C. and at pressures from 1 to 768 mm.

Sargent Column. The rectifying section of a distorted hollow tube column developed by Sargent[40] of the United States Rubber

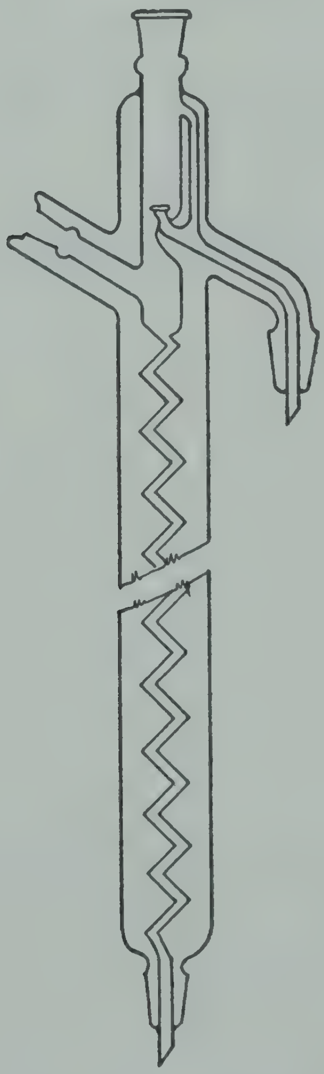

Figure 14. Sargent hollow-tube column.

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[39] Feldman, J., Myles, M., Wender, I., and Orchin, M., *Ind. Eng. Chem.*, **41**, 1037 (1949).

[40] Sargent, H., private communication. Present address: Cal Research, Richmond, California.

Co. General Laboratories[41] is shown in Figure 14. This type of column can be made from several sizes of tubing (7 mm. O.D. to 10 mm. O.D.). Because of the hollow tube design it is very satisfactory for reduced-pressure distillations. Sargent reported that a 7 mm. O.D. column had a rated H.E.T.P. at atmospheric pressure of 3 to 4 in. A similar 7 mm. O.D. column[42] when vacuum-jacketed and equipped with a built-in electronically controlled head has a H.E.T.P. of 1.5 cm. for a 2-foot rectifying section and 1.6 cm. for a 3-foot rectifying section.

The actual assembling of a Sargent rectifying section is simplified if the desired number of sections of tubing are prepared ahead of time by closing off the end and cutting the tubing to the right length, and these sections fused together one piece at a time. Particular attention should be paid to insure that the finished rectifying section is straight vertically, but with each individual section of glass tubing at right angles to the preceding section.

Sargent columns are satisfactory for 1–500 ml. of material at a practical rate of take-off when only a moderate number of theoretical plates are required, and they may be operated from 760 mm. down to 1 mm.

Spinning-Band Columns. Spinning-band columns are especially adapted to vacuum distillations since according to the available data, these columns have the low holdup and the low pressure drop characteristic of a hollow-tube column, but with many times the rectification efficiency. Birch, Gripp, and Nathan[43] describe a spinning band column for high-vacuum work. They reported a pressure drop of 0.05 to 0.06 mm. across the column when the still head pressure was 0.02 mm. of mercury. The normal boil-up rate was 300 ml. per hour but the column showed no signs of flooding at a boil-up rate of 1170 ml. per hour.

Spinning-band columns are relatively simple to build, since the top of the band is fastened to a rod which extends through

[41] United States Rubber Co., Passaic, New Jersey.

[42] Wheeler, E. L., University of California at Los Angeles, unpublished research.

[43] Birch, S. F., Gripp, V., and Nathan, W. S., *J. Soc. Chem. Ind. (London)*, **66**, 33 (1947).

the condenser section and is connected to a stirring motor. The band is usually light and no bottom bearing is necessary. In some instances the band itself extends downwards into the boiling pot and stirs the liquid as a means of reducing ebullition and bumping during distillations conducted at reduced pressures. A modification of this is the attachment of a still pot stirrer to the spinning band. Instructions for constructing a spinning-band column are given by Baker, Barkenbus, and Roswell.[44] A simple spinning-

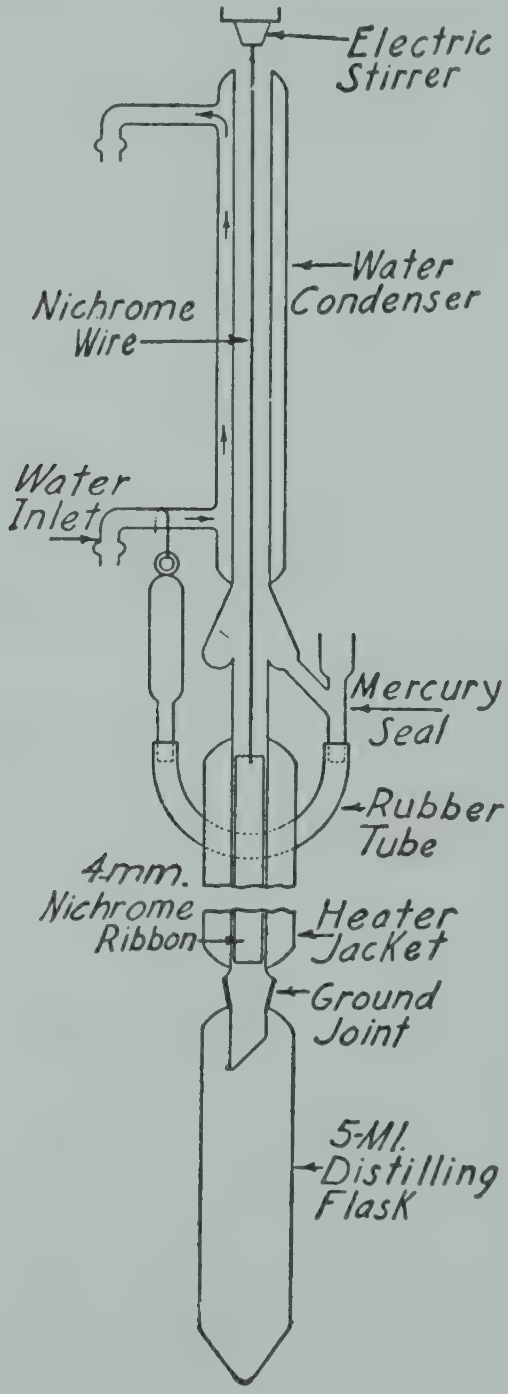
Electric Stirrer
Water Condenser
Nichrome Wire
Water Inlet
Mercury Seal
Rubber Tube
4mm. Nichrome Ribbon
Heater Jacket
Ground Joint
5-Ml. Distilling Flask

Figure 15. Simple spinning-band column. (From A. L. Glasebrook and F. E. Williams in *Technique of Organic Chemistry*, Vol. 4, Interscience, New York, 1951).

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[44] Baker, R. H., Barkenbus, C., and Roswell, C. A., *Ind. Eng. Chem.*, **12,** 468 (1940).

band column is shown in Figure 15. Spinning-band columns are made commercially by H. S. Martin Co.[45] who list a micro spinning-band column with a plate efficiency of 125 theoretical plates and an operating holdup of approximately 0.2 ml. Podbielniak[46] Inc. and the Precision Distillation Apparatus Co.[26] also make spinning-band columns.

Auxiliary Equipment

Figure 16 shows the auxiliary equipment necessary to perform satisfactory distillations under reduced pressure.

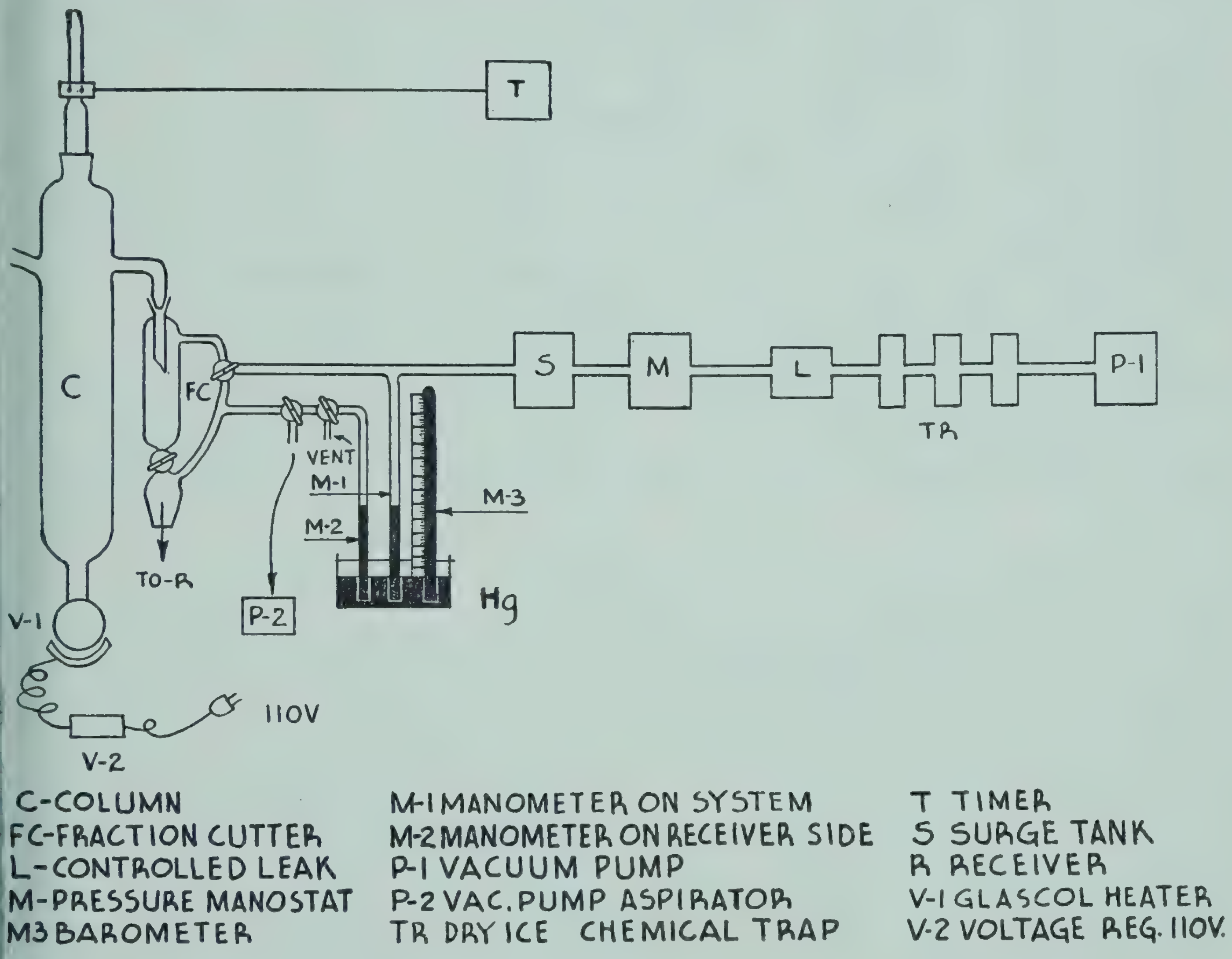
T
C
FC
S
M
L
P-1
TR
VENT
M-1
M-2
M-3
TO-R
P-2
Hg
V-1
V-2
110V
C-COLUMN
FC-FRACTION CUTTER
L-CONTROLLED LEAK
M-PRESSURE MANOSTAT
M3 BAROMETER
M-1 MANOMETER ON SYSTEM
M-2 MANOMETER ON RECEIVER SIDE
P-1 VACUUM PUMP
P-2 VAC. PUMP ASPIRATOR
TR DRY ICE CHEMICAL TRAP
T TIMER
S SURGE TANK
R RECEIVER
V-1 GLASCOL HEATER
V-2 VOLTAGE REG. 110V.

Figure 16. Column assembly for reduced-pressure distillations.

The Vacuum Pump. The vacuum pump should be well protected from stray vapor by both a chemical and dry ice trap (TR in Figure 16), depending on the material being distilled.

Manometer System. Two manometers (M1 and M2 in Figure

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[45] H. S. Martin & Co., Evanston, Illinois.

[46] Podbielniak Inc., Chicago, Illinois.

16) should be arranged in such a way that they may be compared and balanced against each other. One manometer should be on the column side of the system, and the other on the receiver side of the system. By means of equalizing these two manometers a full receiver may be removed, replaced with a new receiver, and re-evacuated to the same pressure as the column before being opened to the column side of the vacuum system. A nearby barometer (M3 in Figure 16) is also a convenience for observing barometric changes; this may be inserted in the same mercury well that supplies the two manometers.

Voltage Control to Boiling Pot. Normal line voltage fluctuation during distillations at atmospheric pressure is not usually great enough to affect the equilibrium of the column. However, even a slight increase in voltage provides a noticeable increase in the boil-up rate of a column operated under reduced pressure. For this reason the voltage should be controlled by means of a voltage regulator. These are commercial items.

Surge Tank. A surge tank will decrease the sensitivity of the column to slight differences in pressure between the column and the receiver during the changing of receivers. The surge tank, to

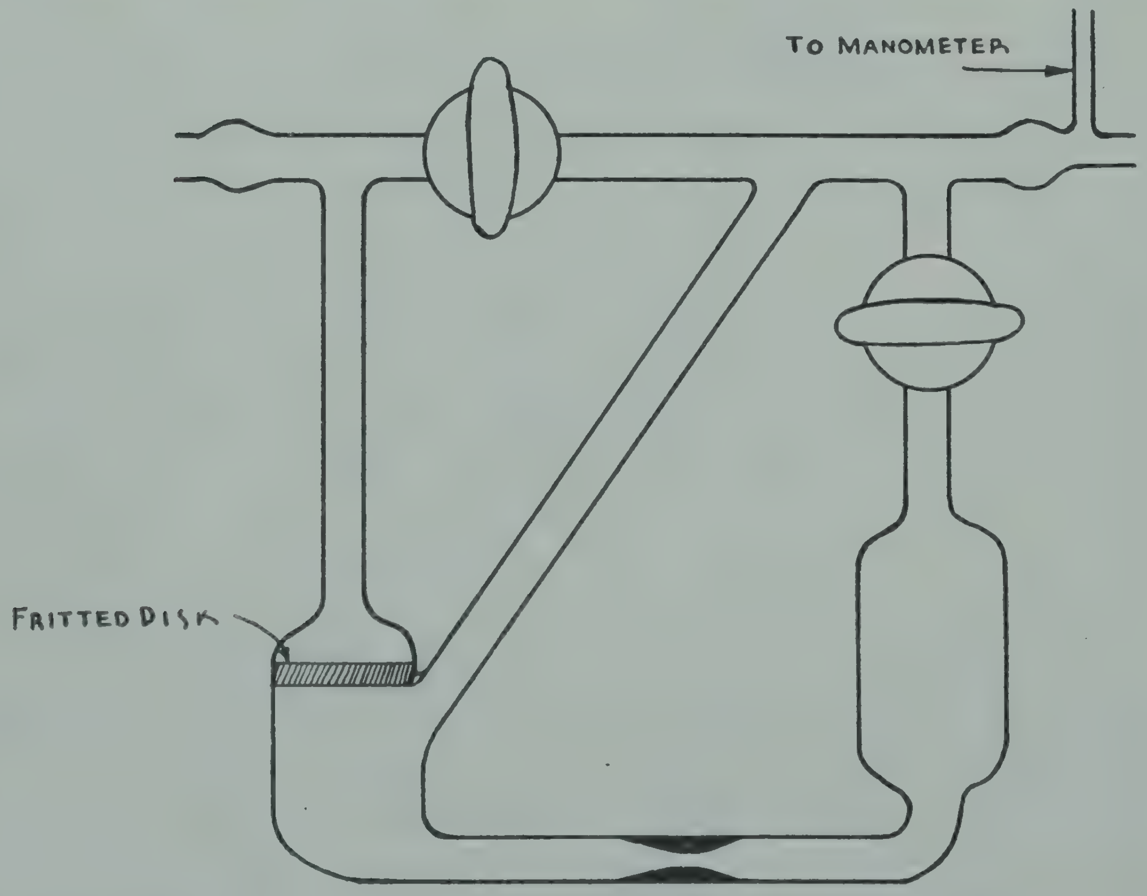

Figure 17. Fritted glass pressure manostat.

be the most useful, should have a capacity of 1 to 2 liters.

Controlled Leak. Many devices may be used for small controlled leaks. A simple one which gives satisfactory results is the base of an ordinary Bunsen burner. The needle valve which

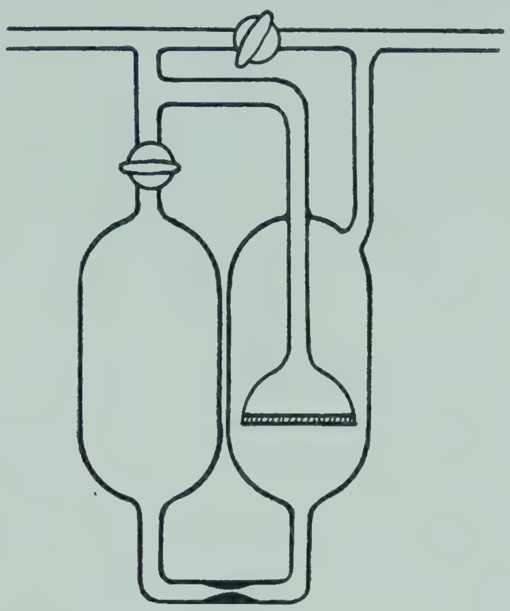

Figure 18. Fritted glass pressure manostat.

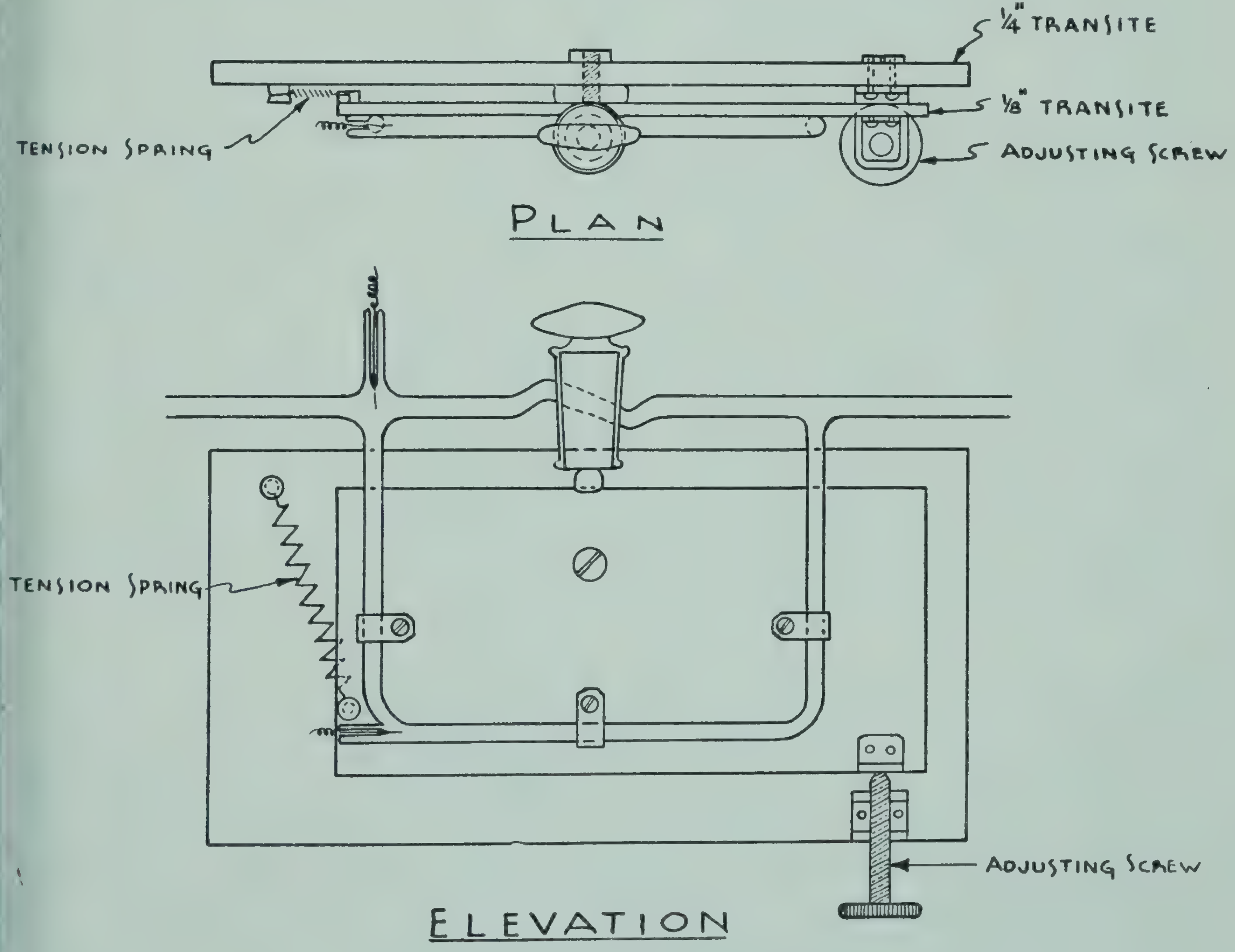
¼" TRANSITE
⅛" TRANSITE
ADJUSTING SCREW
TENSION SPRING
PLAN
TENSION SPRING
ADJUSTING SCREW
ELEVATION

Figure 19. Mercury manostat for electronic control.

regulates the flow of gas into the burner may be used to regulate the amount of air leaking into the vacuum system. The burner may be inserted in the vacuum line by means of a glass T and rubber tubing.

Pressure Regulators. Two types of glass sintered manostats are shown in Figures 17 and 18. During operation of these manostats, air is allowed to escape through the fritted plate when the mercury level drops. For some pressures these manostats will provide sufficient control without an additional controlled leak. For most purposes, however, it is advisable to install a separate control leak in the line.

In Figure 19 a mercury manostat is shown. When this is used in conjunction with an electronic power supply, it gives satisfactory control of the pressure.

All three of the regulators described are available commercially.[26]

Preliminary Operational Procedure

Reduction of the pressure employed in a distillation involves several changes from the operational procedure followed for atmospheric distillation. Before any vacuum distillation is attempted, all stopcocks, ground joints, and hose connections should be thoroughly lubricated with a vacuum-type stopcock grease. The complete system should be thoroughly checked for leaks and the mechanical pump tested to insure its pumping the required vacuum. In addition, the pump should be adequately trapped to protect it from stray vapors.

Degassing the Sample

Since most organic liquids are saturated with air, the charge for a vacuum distillation should be degassed before heat is turned on full under the kettle. This degassing is accomplished by simply reducing the pressure in the distillation apparatus after the sample has been placed in the kettle. With highly viscous materials, slight heating may be necessary to make the charge fluid enough to release the air. No rise in pressure when the vacuum pump is turned off will indicate that degassing is complete. Liquids which foam must be degassed carefully to keep the foam from carrying away the sample.

Adjusting Boil-up Rate

A rule which will generally produce satisfactory results is as follows: The column capacity (throughput) varies as the square root of the distillation pressure. For example, since a sample which boils at 60 ml. per hour at total reflux and atmospheric pressure should distill at approximately one-fourth this rate at 40 mm., approximately one-fourth of the heat input is required.

Elimination of Bumping

A satisfactory antibumping procedure is to fuse sintered glass to the bottom of the boiling flask. This is done by putting a small amount of ground glass (50- to 60-mesh) in the empty flask and then heating the bottom until the flask softens sufficiently for the ground glass to adhere to the surface. The glass should not be heated long enough to fuse the ground glass into the flask too well, defeating the purpose for which it was intended. The flask should be thoroughly annealed before any distillations are attempted.

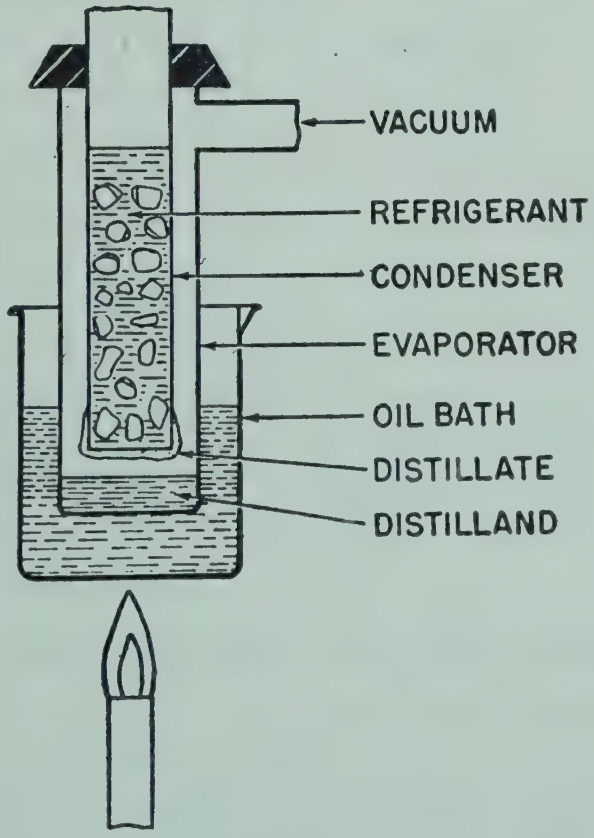

Figure 20. Simple molecular still. (From E. S. Perry in *Technique of Organic Chemistry*, Vol. 4, Interscience, New York, 1951.)

Another method for preventing bumping is to introduce a small piece of a broken glass fritted plate; lumps of pumice stone, coal, or a piece of a porous pot can also be used. These are effective down to a few millimeters of mercury as long as boiling is maintained. The pieces of fritted glass can be dried and re-used.

A third type involves the use of a material not wetted by the material being distilled. Teflon (polytetrafluoroethylene), which is very unreactive chemically and withstands temperatures up to 200° C., is probably the most useful of these. Since this material is not wetted by most liquids, it maintains a thin film of air over its entire surface when immersed in liquid. It is from this film of air that the bubble forms during heating of the liquid.

MOLECULAR DISTILLATION

Quite often it is necessary to purify a high-boiling material which is unstable at its boiling point. This is best done under high vacuum in specially designed molecular stills. Molecular distillation is not the ideal method of separating materials since it gives a poor separation. It is, however, a means of distilling many substances which cannot be readily purified any other way.

In molecular distillations, the majority of molecules distilling from the liquid pass directly to the condenser. For this reason the condenser is placed in such a position that the distance from the surface of the charge to the condenser is less than the mean free path of the distilling molecules, that is, less than the average distance traveled by a molecule between collisions.

Figures 20 and 21 give the plans for two satisfactory molecular stills. A complete treatise on molecular distillation theory and practice is recommended reading.[47]

AZEOTROPIC DISTILLATION

The separation of some liquid mixtures is made difficult because they distill at a constant temperature under a constant

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[47] *Technique of Organic Chemistry*, Vol. 4, p. 495, Interscience, New York, 1951.

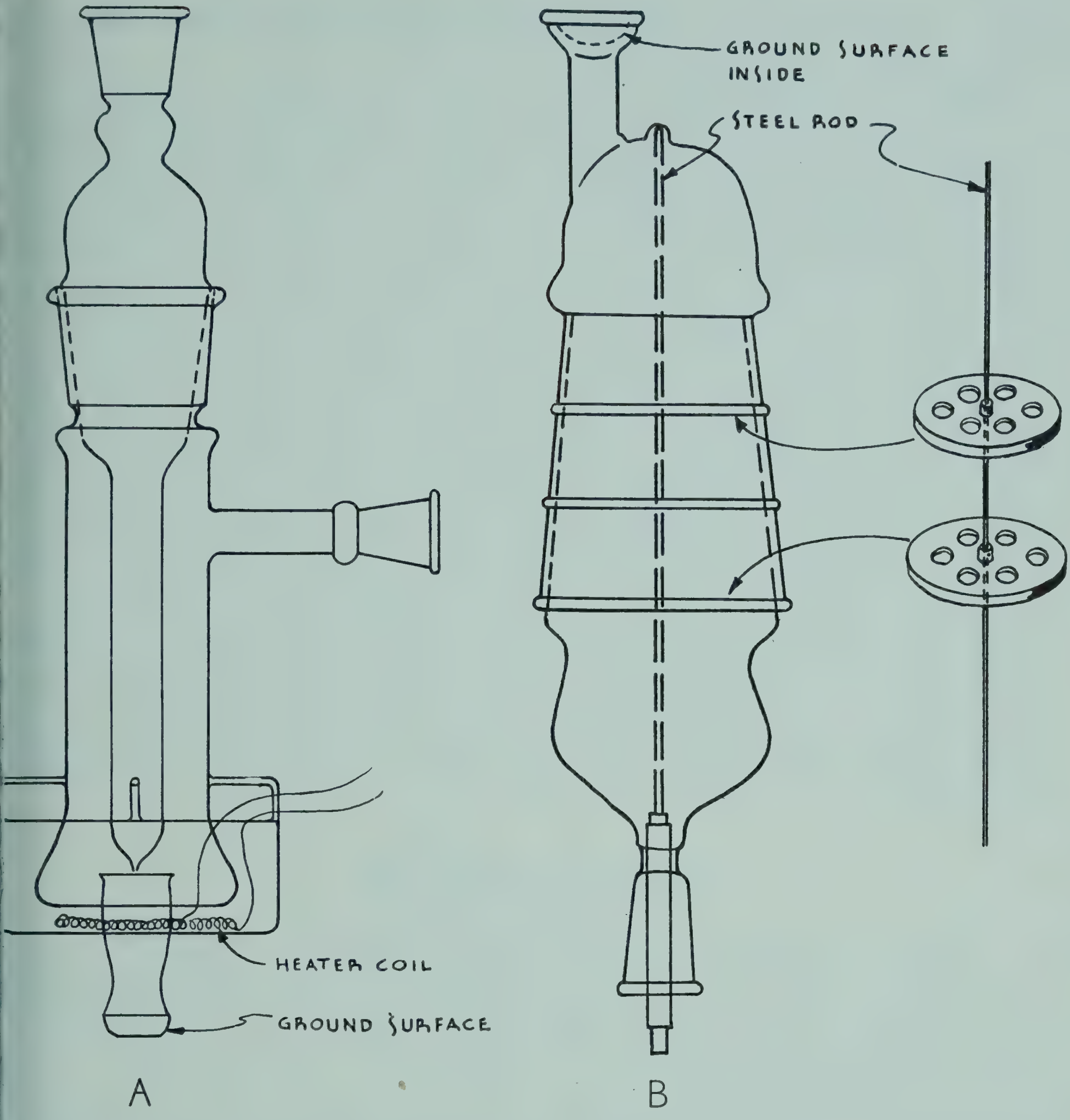

Figure 21. A. Molecular still. B. Receiver for molecular still.

pressure and without any change in composition. Such a mixture, called an azeotropic mixture, usually has a boiling point higher or lower than either of its pure components. A constant-boiling mixture (CBM) is another name for an azeotropic mixture. In some instances, separation of an azeotropic mixture is simplified by phase separation on cooling, that is, the azeotrope separates into two layers on cooling.

A second method of separating an azeotrope is "salting out", in which a salt such as sodium or calcium chloride is added to the mixture, causing phase separation by salting out. Sometimes even adding an excess of one component of the azeotrope will cause phase separation.

Chemical removal of the entrainer is sometimes possible when the recovery of the entrainer is essential. In an azeotropic mixture of hydrocarbons and organic acids, the latter may be removed by washing with alkali, while amines, such as aniline, may be removed with mineral acids. In solvent extraction or water washing, the entrainer is recovered by liquid extraction with water.[48]

Some azeotropes are separated by distilling under reduced pressure. For example, nearly all the azeotropes formed by alcohols are separated by distilling at 70 mm. pressure or less. Azeotropes are frequently separated by a second distillation with a different entrainer which forms an azeotrope from which it may be more easily recovered. A partial reflux head employing a vapor take off is particularly useful for an azeotropic distillation which forms more than one liquid phase. With this kind of mixture a liquid take-off is often unsatisfactory because the liquid will separate in the trap formed above a stopcock.

USE OF A CHASER

When the amount to be distilled is small, or during analytical distillations when time is at a premium, a small sample may be run with the aid of a chaser. By this means samples hardly larger than the column operating holdup may be fractionated. To be satisfactory, chasers should be chemically inert and inexpensive, and then should not form azeotropic mixtures. In addition, they should be at least 20° C. higher boiling than the end point of the material being distilled. Another use for chasers is in distillations where it is desirable to have complete fractionation (provided the holdup or residue is volatile).

The following chasers are generally used:

Cymene	175° C. b.p.
Diphenyl	254° C.
Acenapthene	277° C.
Phenanthrene	340° C.

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[48] "Distillation under High Vacuum" in *Trans. Am. Inst. Chem. Engrs.* **41**, 327 (1945).

LOW-TEMPERATURE DISTILLATIONS

When a mixture of gases is to be distilled they are usually liquefied and distilled by what is commonly referred to as a low-temperature distillation. Low-temperature distillations are defined as distillations below room temperature as against high-temperature distillations, which are those above room temperature. In the field of low-temperature distillations Podbielniak[49] has been one of the outstanding contributors. The early Podbielniak[50] low-temperature fractional distillation consisted of a vacuum-jacketed column with an inner tube of about 3.8 mm. I.D. × 77 cm long. Expansion between the inner and outer members of the column was compensated for by a bow in the inner tube. The packing for these columns consisted of a simple wire-coil. In some cases the coil diameter was slightly less than the inside diameter of the column tube. Double coils were also made with the inside coil wound in reverse of the outer coil. See Figure 22. The H.E.T.P. of simple wire-coil packing as reported by Podbielniak[51] was approximately 2 in. Rose[52] reported that the H.E.T.P. of simple wire-coil packing varied widely according to the vapor velocity.

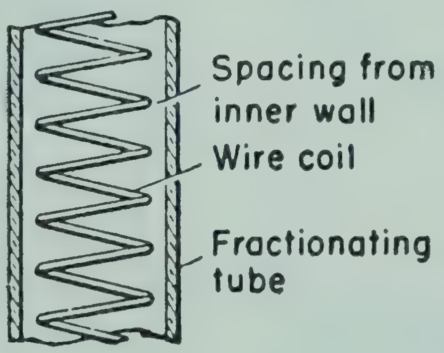

Figure 22. Simple wire-coil packing. (From A. L. Glasebrook and F. E. Williams in *Technique of Organic Chemistry*, Vol. 4, Interscience, New York, 1951.)

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[49] Podbielniak, W. J., Podbielniak Inc., Chicago, Illinois.

[50] Podbielniak, W. J., *Ind. Eng. Chem. Anal. Ed.*, **3**, 180 (1931). U.S. Patent 2,009,714.

[51] Podbielniak, W. J., "*Apparatus and Methods for Precise Fractional Distillation Analysis*," meeting Am. Chem. Soc., Atlantic City, 1941.

[52] Rose, A., *Ind. Eng. Chem.*, **28**, 1210 (1936).

In 1941 Podbielniak[53] described a new precision-spaced wire packing called Heli-Grid with a H.E.T.P. of less than 0.5 cm., and a new model low-temperature column called the Super-cal column which was designed to provide a fully adiabatic column for the entire temperature range of —180° to 300° C. In this column it is possible to distill a sample ranging in composition from methane to lubricating oil, beginning with a reflux temperature of —161.4° C. at atmospheric pressure and ending with 300° C. in the reflux at 20 mm. absolute pressure.

Heli-Grid packing consists of a wire-coil wound around a central solid core and around each other in telescoping layers as shown in Fig. 23. The packing was named Heli-Grid, according

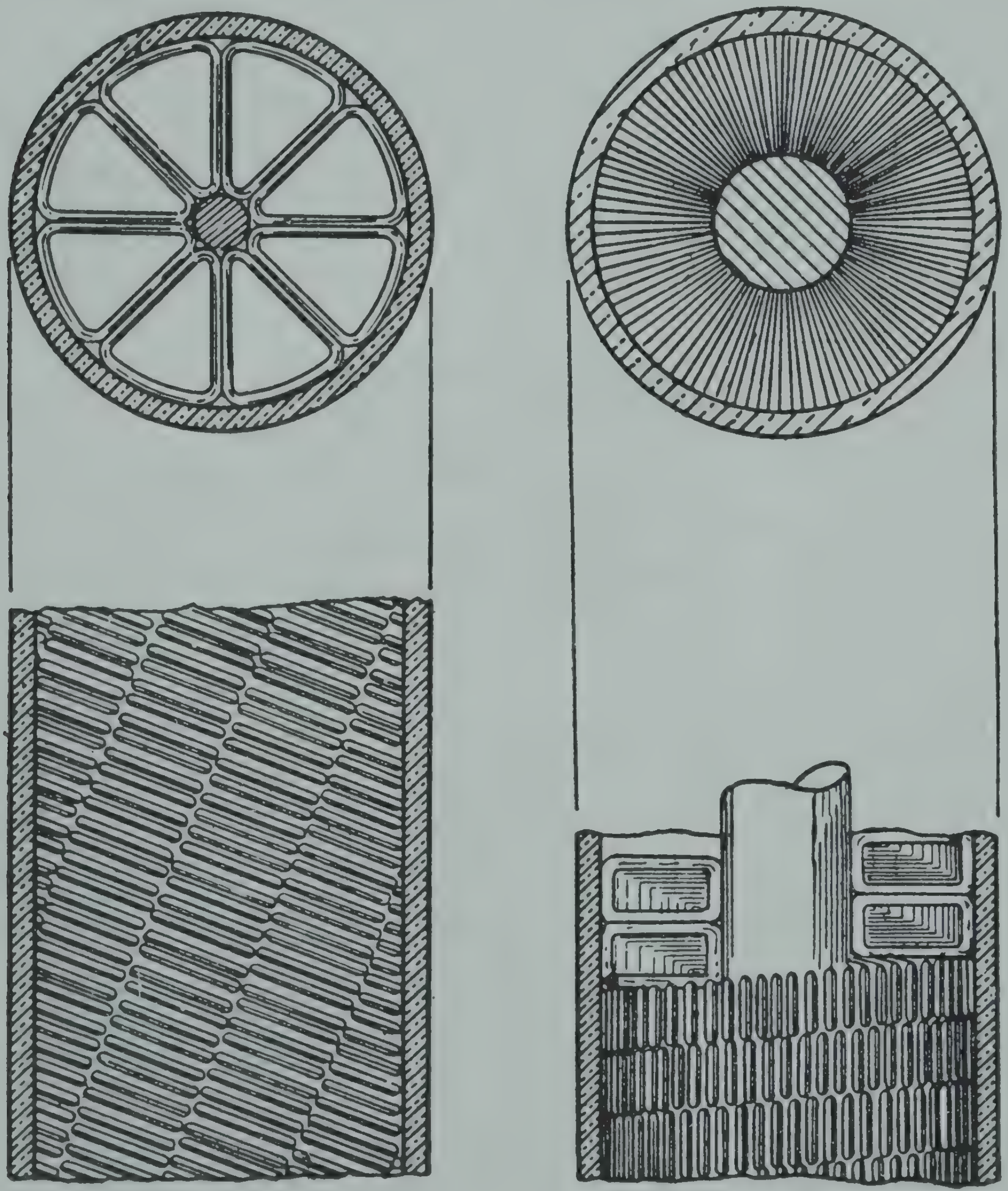

Figure 23. Heli-Grid packing. (From A. L. Glasebrook and F. E. Williams in *Technique of Organic Chemistry*, Vol. 4, Interscience, New York, 1951.)

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[53] Podbielniak, W. J., *Ind. Eng. Chem.*, **13**, 639 (1941).

to Podbielniak, because of its resemblance to helical grids. Because of the close winding and resulting capillary action of the coils, it is claimed that a liquid will actually follow the capillary wire coils instead of channeling even if the packing is tilted nearly horizontal before the liquid is poured into the column.

GENERAL DESCRIPTIONS OF FRACTIONATING EQUIPMENT

Fractionating Column

In Figure 24 is shown one of the more simple types of distillation assemblies. After the sample is brought to the boiling point by means of a Bunsen burner, the vapor passes off and is condensed in the sloping water condenser, from which it drops into the receiver. Figure 25A shows a Vigreux column suitable for micro distillations, and Figure 25B an exploded view of the receiver.

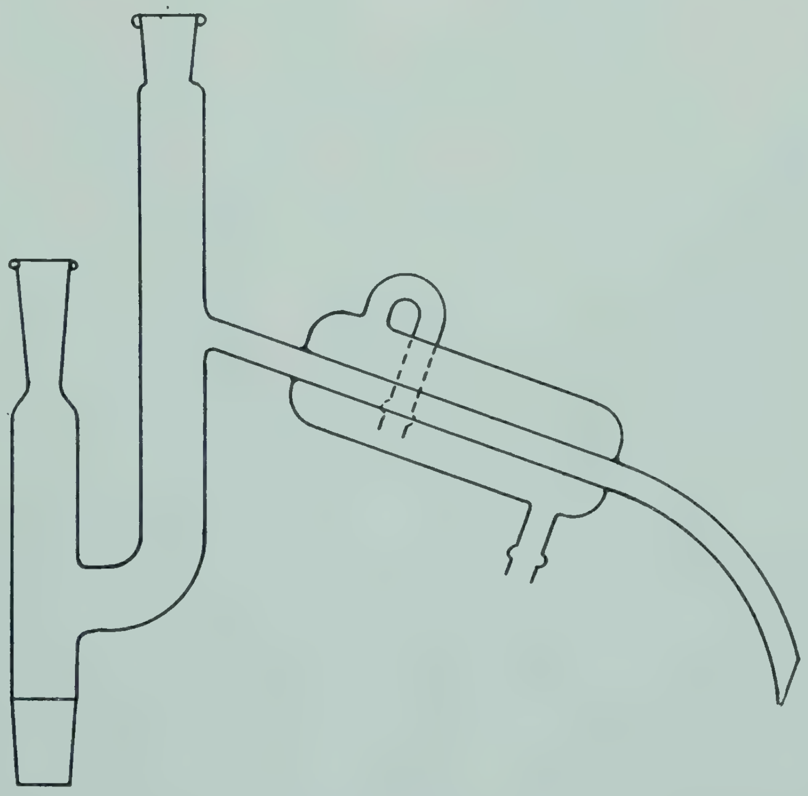

Figure 24. Simple distilling unit.

Figure 26 shows a column in which the tube forming the rectifying section is lined with a coating of ground glass. This section is prepared as previously described on page 248 or it may be prepared by supporting an iron rod concentrically in a section of glass tubing the desired size, and packing ground glass into the open space between the metal rod and the inner wall of the glass tubing. The diameter of the iron rod should be about half the diameter of the glass tube. The filled section of tubing should then be heated in an oven to 800° to 900° C. for about 15 minutes to fuse the ground glass to the wall of the glass tube, after which it is allowed to cool slowly. When the glass has cooled to room temperature the metal rod may be removed either by simply pulling it out or by dissolving it with acid. A column as shown, with a rectifying section 60 cm. long and of 7 mm. I.D. tubing, will be satisfactory for charges from 25 to 300 ml. The theoretical plate rating will be about 5.0 and the H.E.T.P. about 12 to 12.5 cm.

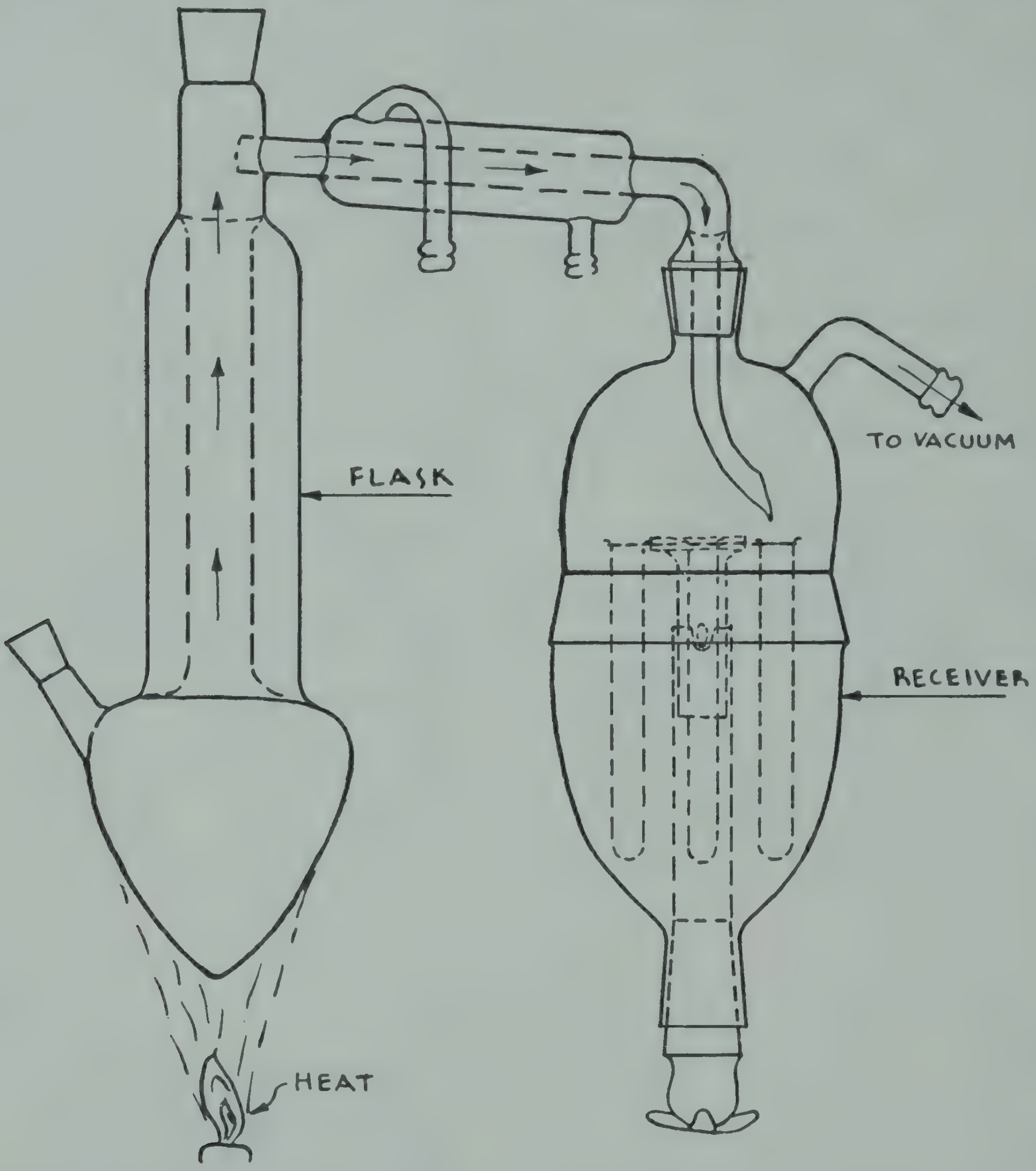

Figure 25A. Vacuum-jacketed Vigreux column and receiver.

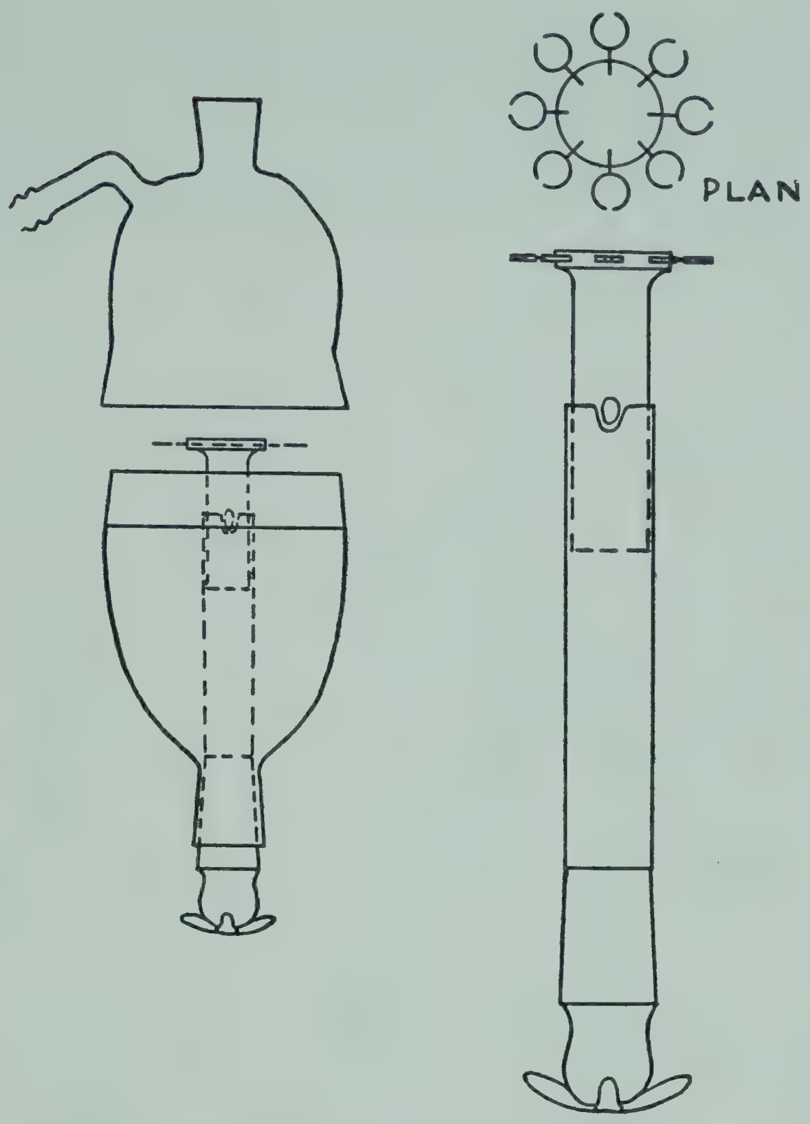

Figure 25B. Exploded view of receiver.

The column shown in Figure 27 can be made with a rectifying section of almost any desired length or inside diameter. The head may be removed for easy cleaning and for changing the packing. These columns are usually silvered with a strip window running the entire length for viewing the packing and observing operating conditions. Most of the still heads shown (pp. 295—302) may be used with this column.

Figure 28 shows a column for random-type packing with a manually controlled take-off and a removable condenser section. This column can be made with different lengths and inside diameters for the rectifying section.

A column with an electronically controlled take-off is shown in Figure 29. This is a commercial model as manufactured by the Precision Distillation Apparatus Co.[26] and comes in 16 variations of lengths and diameters.

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[26] Precision Distillation Apparatus Co., 2304 Wilshire Blvd., Santa Monica, California.

The column shown in Figure 30 is similar to the column in Figure 26, except that the rectifying section is empty, allowing the column to be packed with any desired random-type packing.

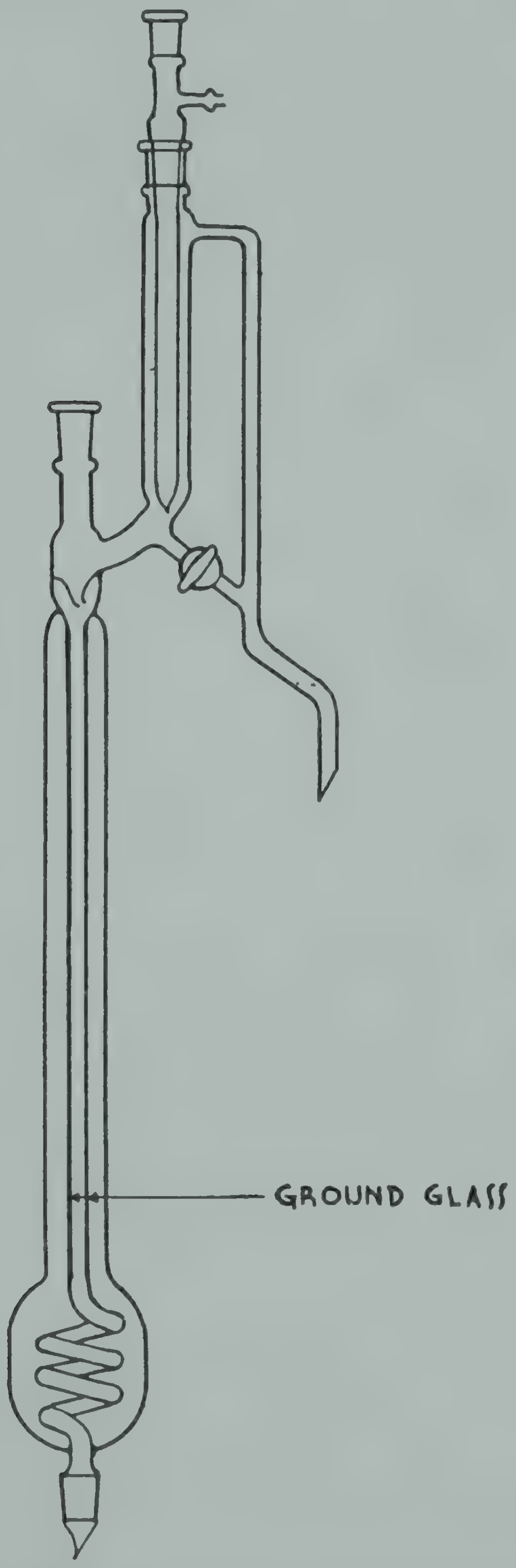


Figure 26. Vacuum-jacketed column with ground-glass rectifying section.

At times it is desirable to mount two or more vacuum-jacketed sections like the column in Figure 27, together to give the increased efficiency of a longer column. The rigid mounting of

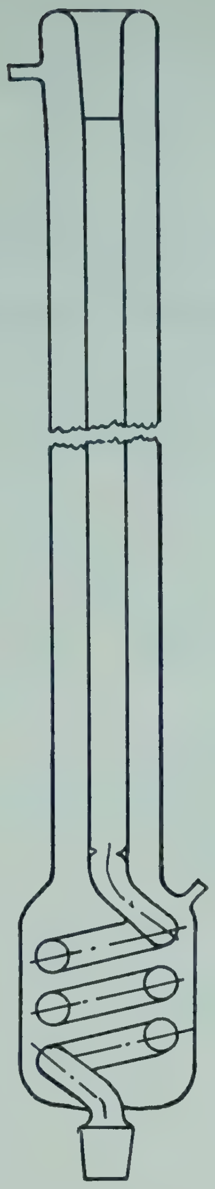


Figure 27. Column for random-type packing.

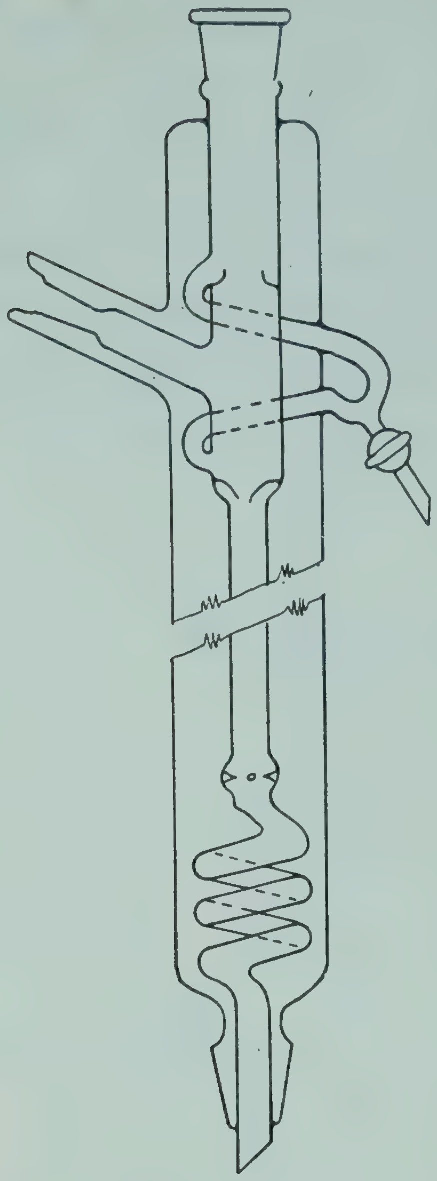


Figure 28. Manually controlled column for random-type packing.

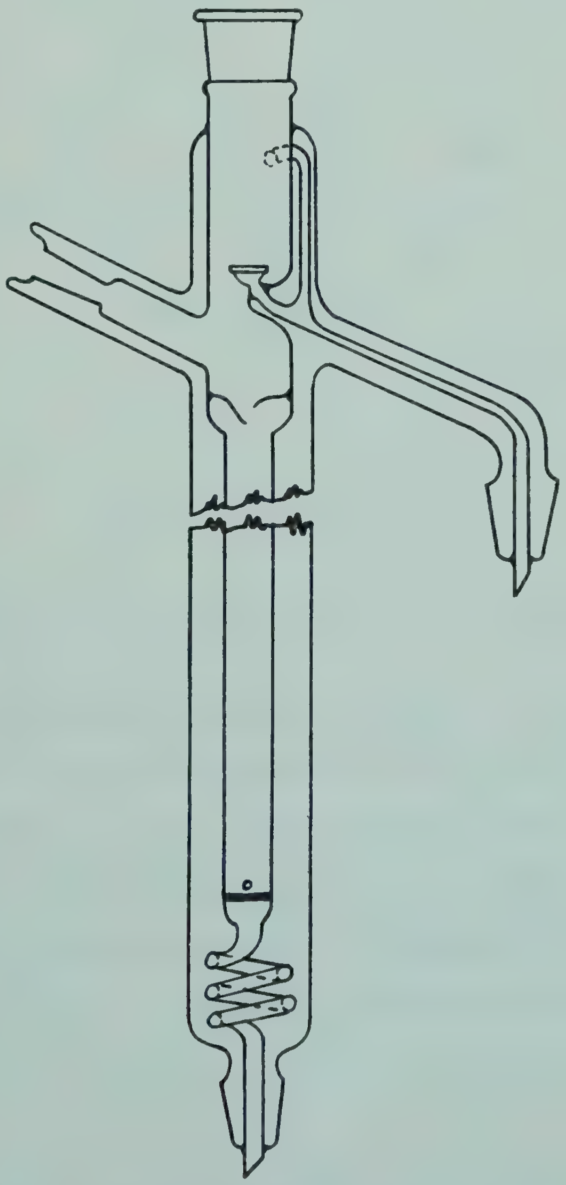


Figure 29. Electronically controlled column for random-type packing.

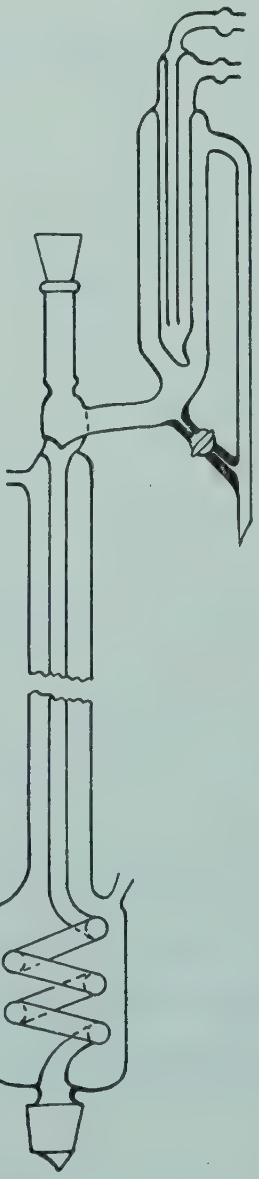
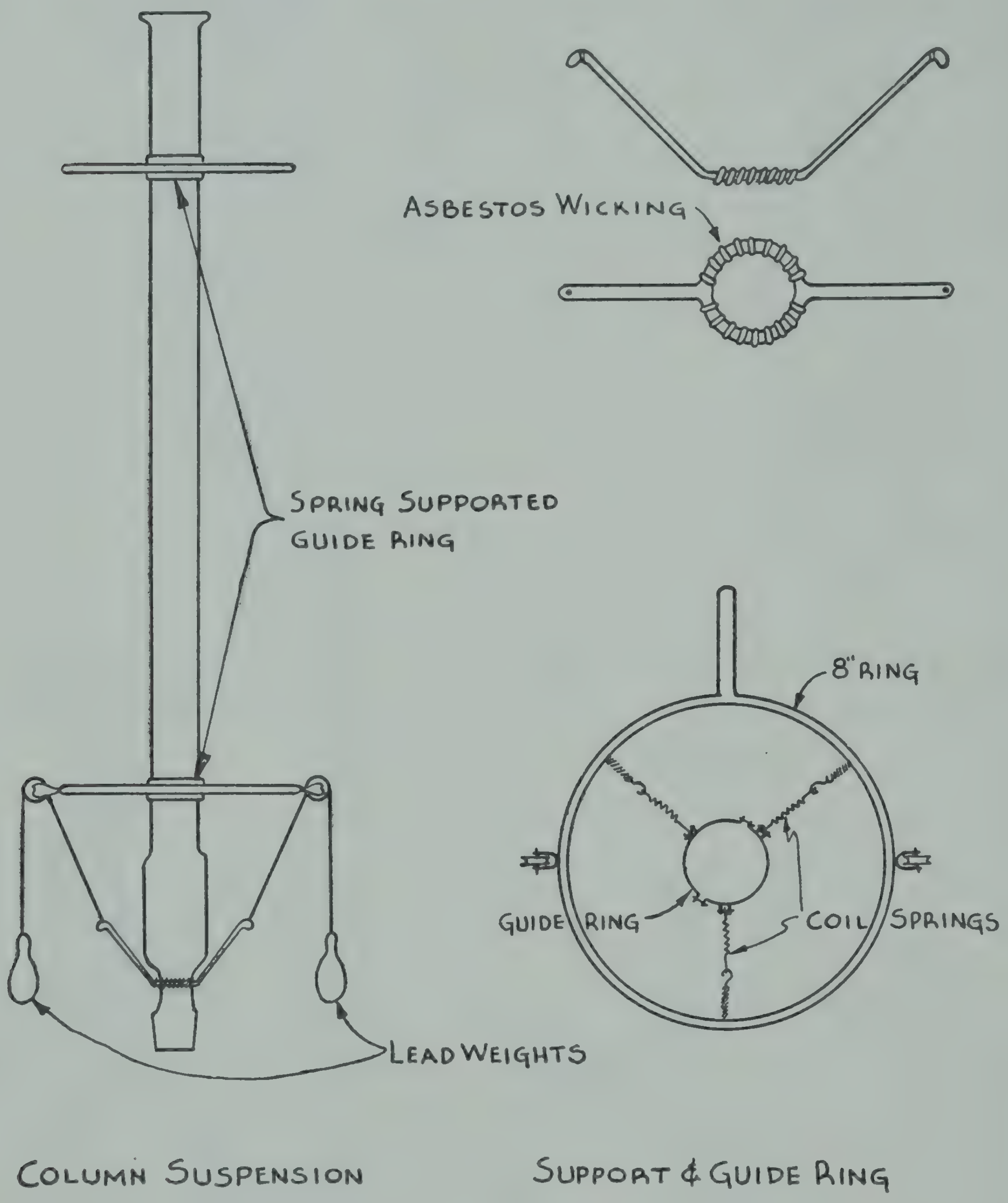


Figure 30. Column for random-type packing.

several sections together presents a problem which may be solved by using the spring suspension shown in Figure 31. Two spring guide rings should be made for each column section and if a large type of still head is to be used, one spring guide ring should be provided for it. The lead weights should be sufficient to balance the weight of the column assembly, thus removing the weight of the columns from the springs in the guide ring. The sole purpose of the springs is to center the assembly. When several sections are used, it is advisable to use separate lead weights and balance each section individually.


Asbestos Wicking
Spring Supported Guide Ring
8" Ring
Guide Ring
Coil Springs
Lead Weights

Column Suspension

Support & Guide Ring

Figure 31. Spring tension mounting for fractionating columns.

Still Heads

The still heads shown in Figures 32 and 33 can be used in any column with matching $ joints and for distillations in which stopcock control of the take-off is sufficiently accurate. The take-off may also be regulated by leaving the stopcock wide open and rotating the cold finger condenser to divide the amount of distillate entering the take-off line and that returning to the column as reflux. A vacuum manifold makes either head suitable for atmospheric or reduced-pressure distillations.

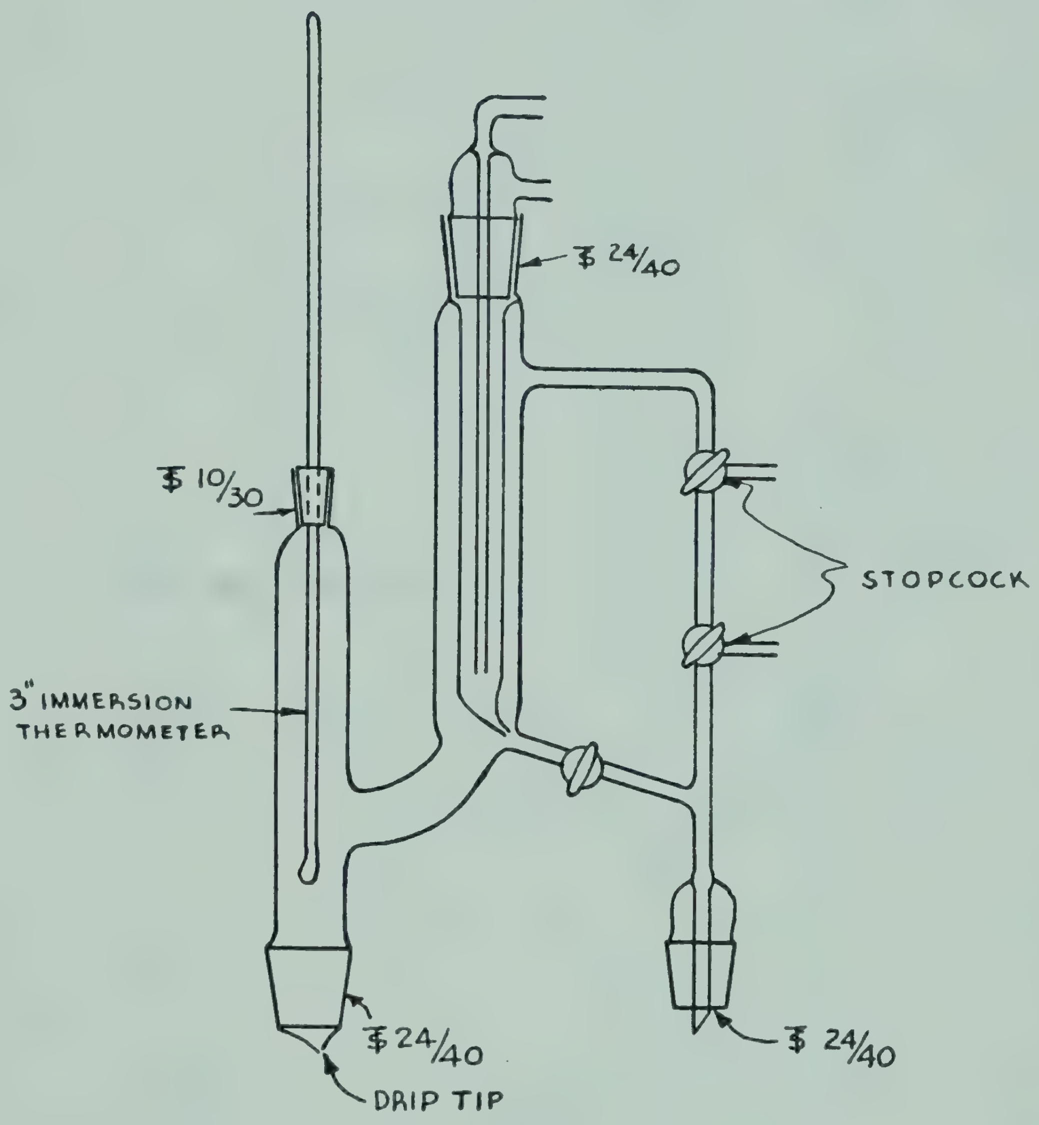


Figure 32. Still head.

The still head shown in Figure 34 is suitable for moderate low-temperature distillations. With a suitable coolant in both traps, complete condensation of the vapor is insured, especially if the condensate is collected in a cooled fraction collector.

High-temperature distillations may be carried out using the still heads shown in Figure 35A. The vacuum jacket should be fitted with enough expansion bellows to compensate for the temperature to which the head will be subjected. The still head shown in Figure 35B is a vacuum-jacketed vapor take-off head. It should have sufficient expansion bellows to compensate for the temperature at which it will be used.

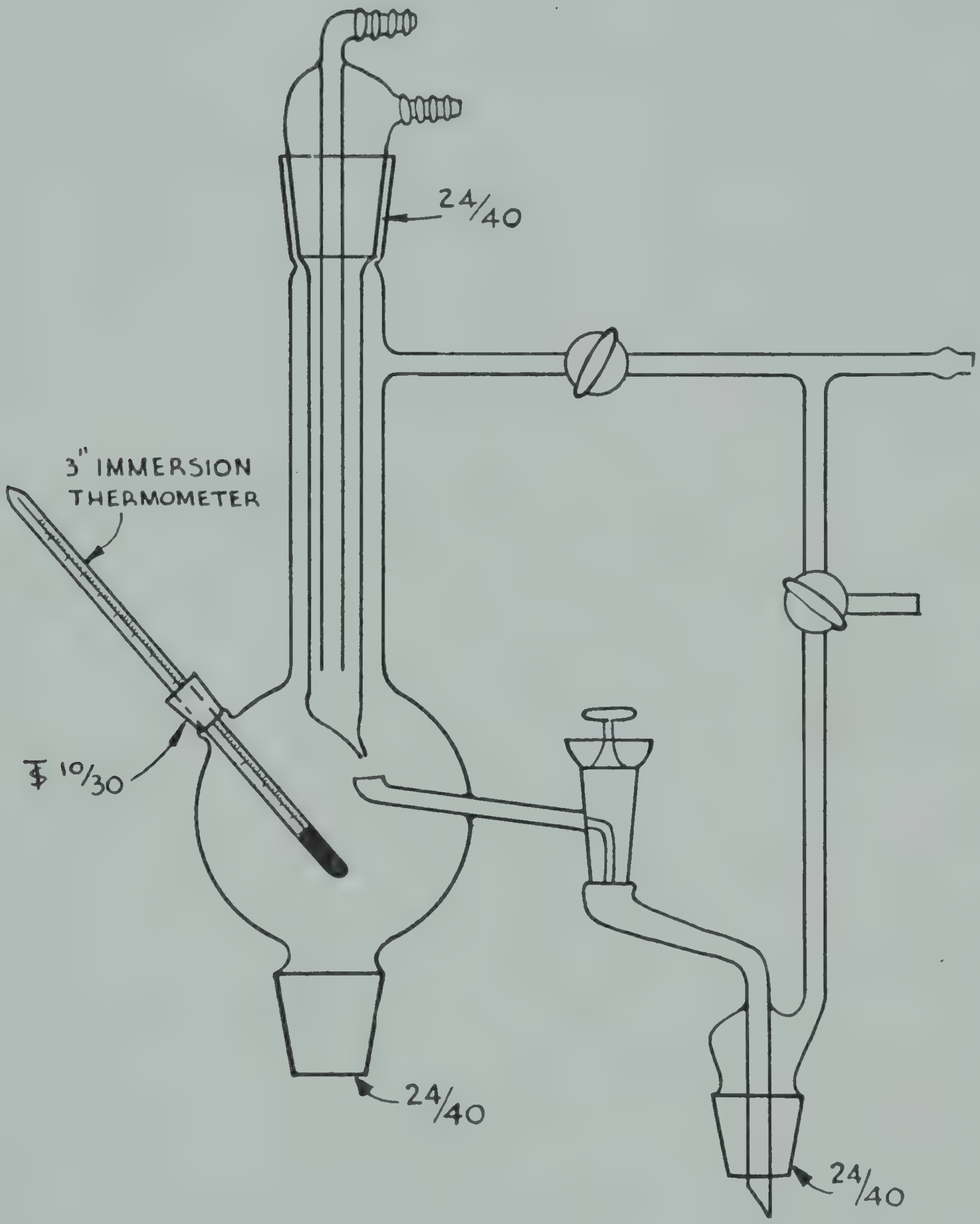


Figure 33. Still head.

A still head with automatically controlled take-off is shown in Figure 36. This head is completely vacuum-jacketed and thus suitable for either high- or low-temperature distillations. An equalizing line, inside the vacuum jacket, between the take-off line and the top of the head provides for satisfactory operation at reduced pressure. For low-temperature distillations the standard

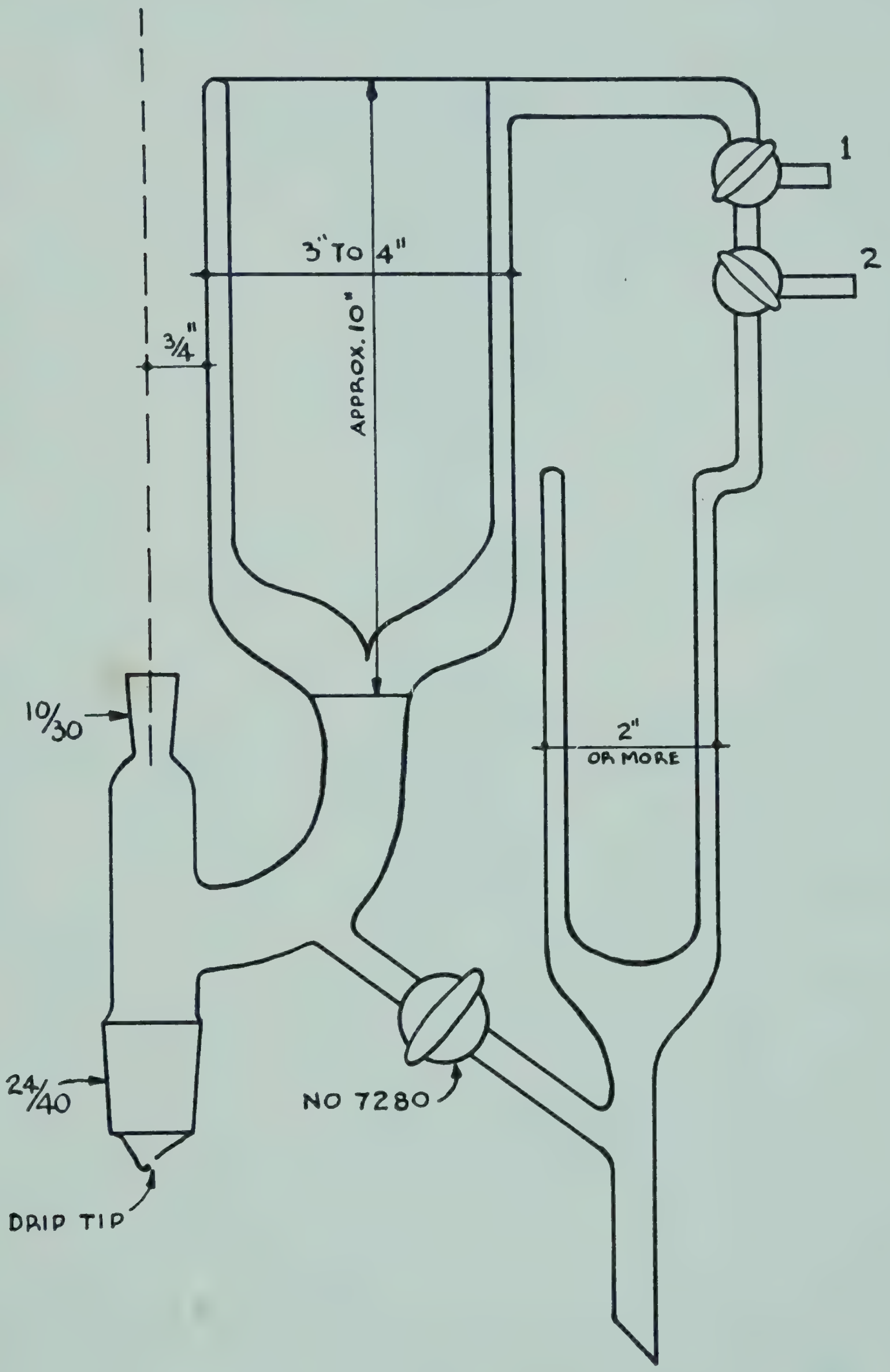


Figure 34. Still head for moderately low-temperature distillations.

removable condenser of this head is replaced by the vacuum-jacketed cold finger shown in Figure 37. This cold finger is also interchangeable with the coil condenser of the concentric-tube column in Figure 10 for reduced-temperature distillations.

A second type of automatic liquid dividing head is shown in Figure 38, and an automatic vapor dividing head is shown in Figure 39.

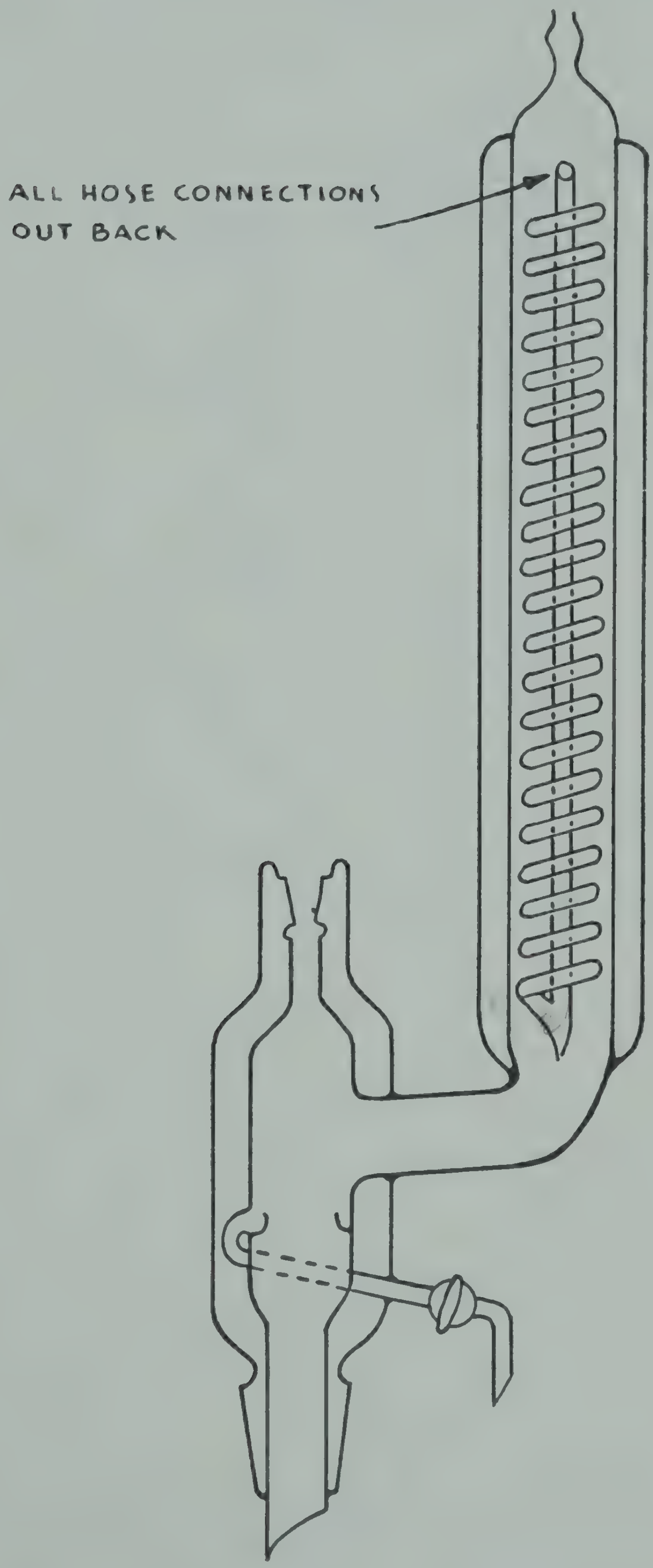


Figure 35A. High-temperature distillation still head.

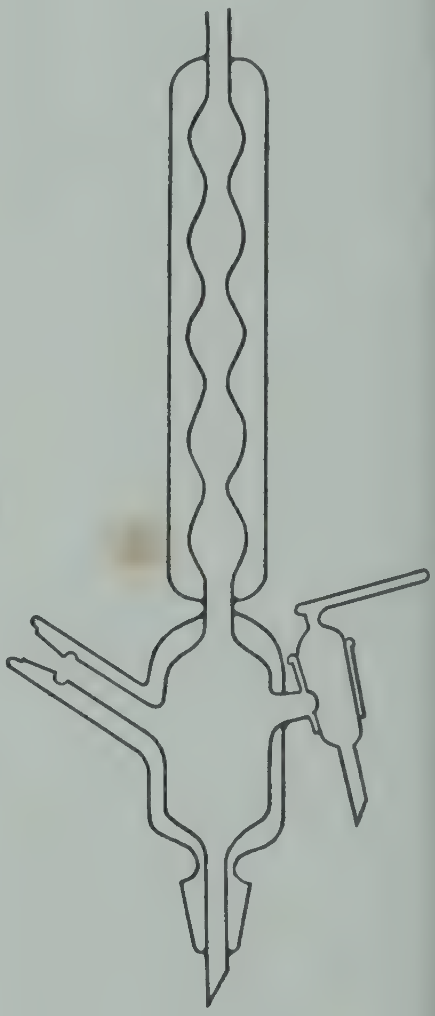


Figure 35B. High-temperature vapor take-off still head.

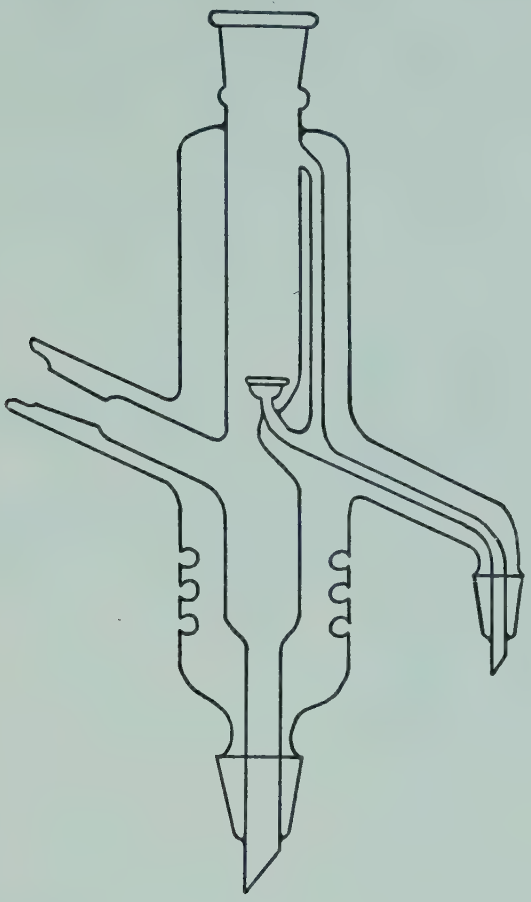


Figure 36. Electronically controlled, vacuum-jacketed still head.

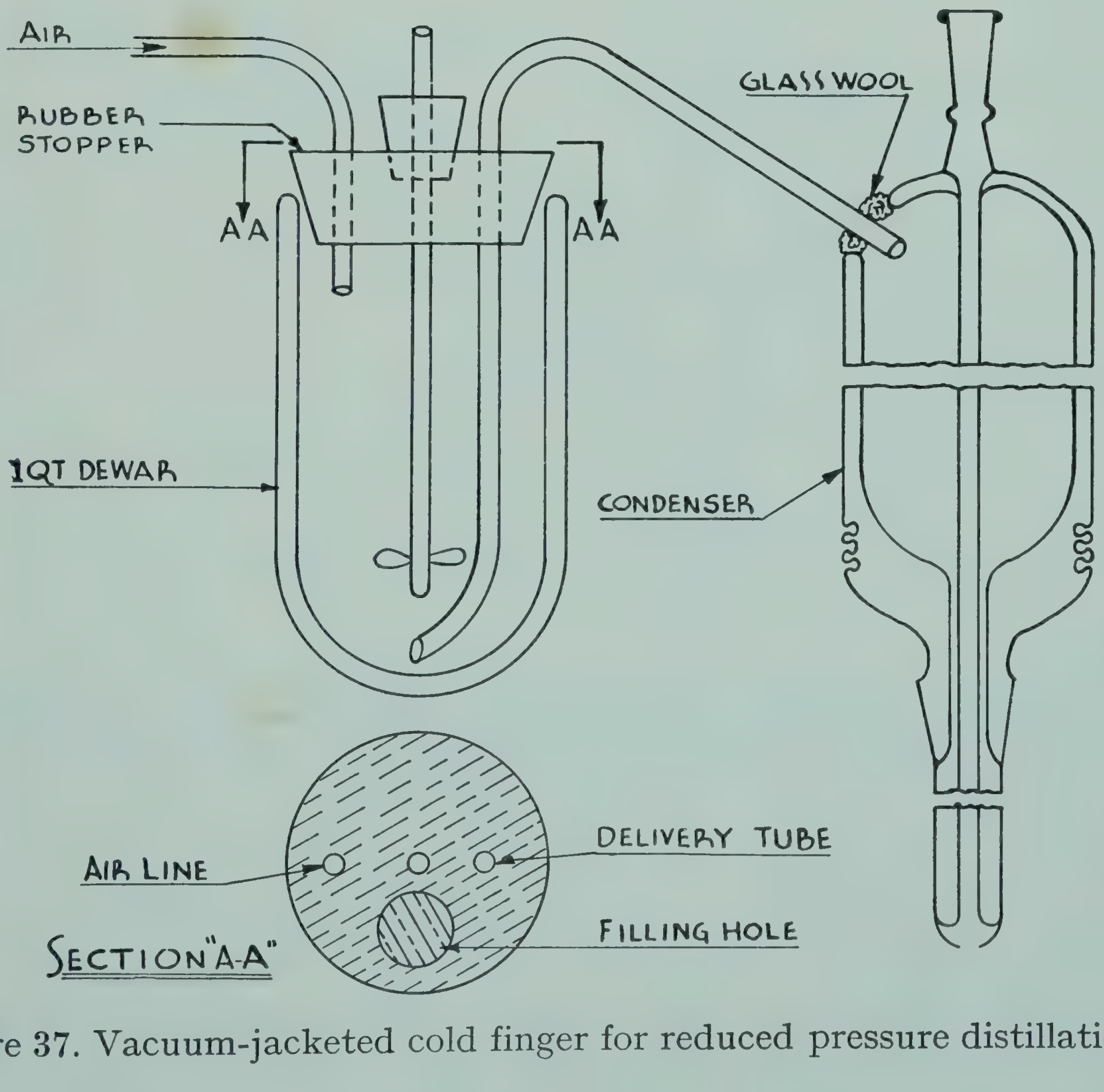

AIR
RUBBER STOPPER
A A
A A
GLASS WOOL
1QT DEWAR
CONDENSER
DELIVERY TUBE
AIR LINE
FILLING HOLE
SECTION "A-A"

Figure 37. Vacuum-jacketed cold finger for reduced pressure distillations.

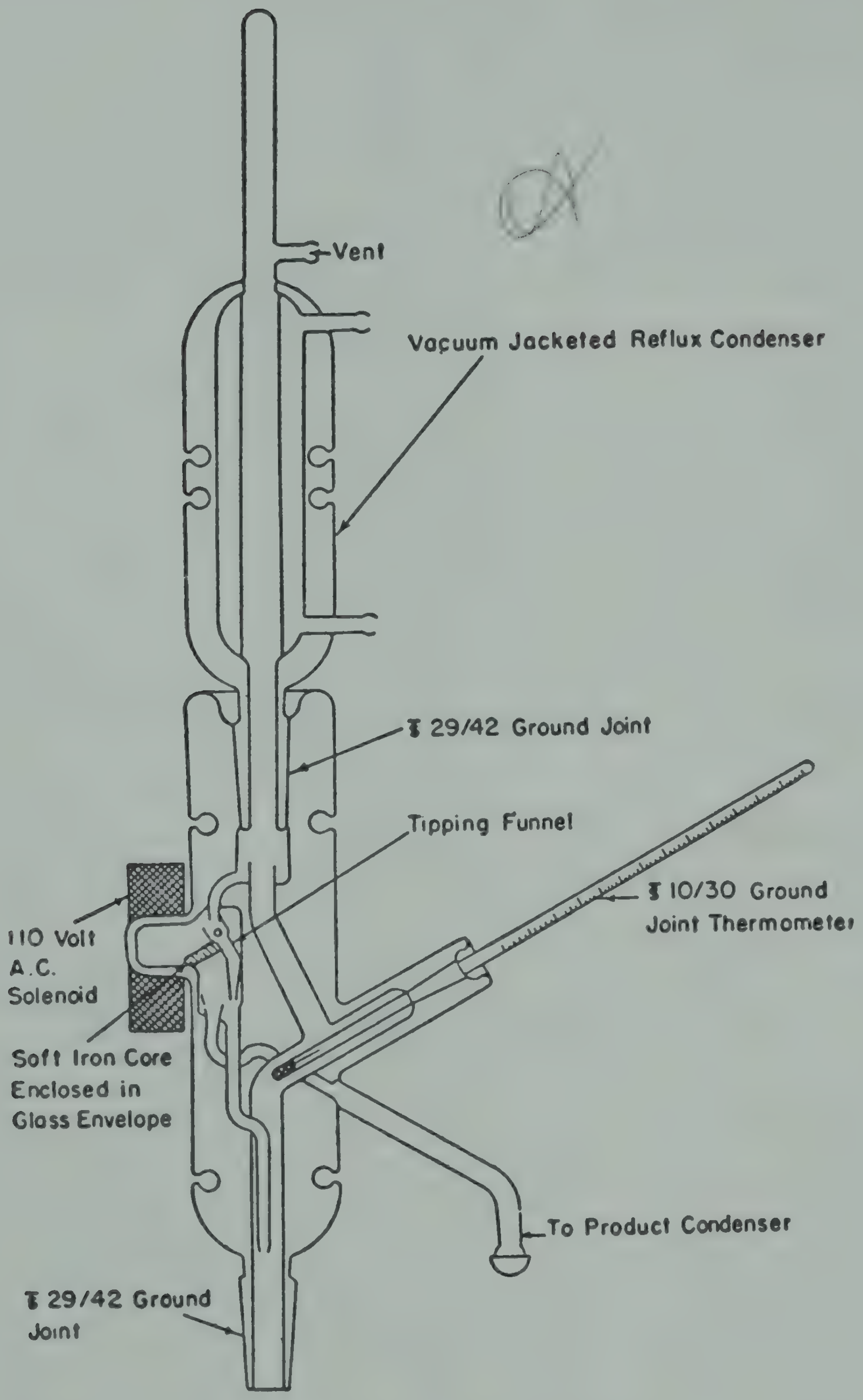

Vent
Vacuum Jacketed Reflux Condenser
₮ 29/42 Ground Joint
Tipping Funnel
₮ 10/30 Ground Joint Thermometer
110 Volt A.C. Solenoid
Soft Iron Core Enclosed in Glass Envelope
To Product Condenser
₮ 29/42 Ground Joint

Figure 38. Automatic liquid dividing still head. (From A. L. Glasebrook and F. E. Williams in *Technique of Organic Chemistry*, Vol. 4, Interscience, New York, 1951).

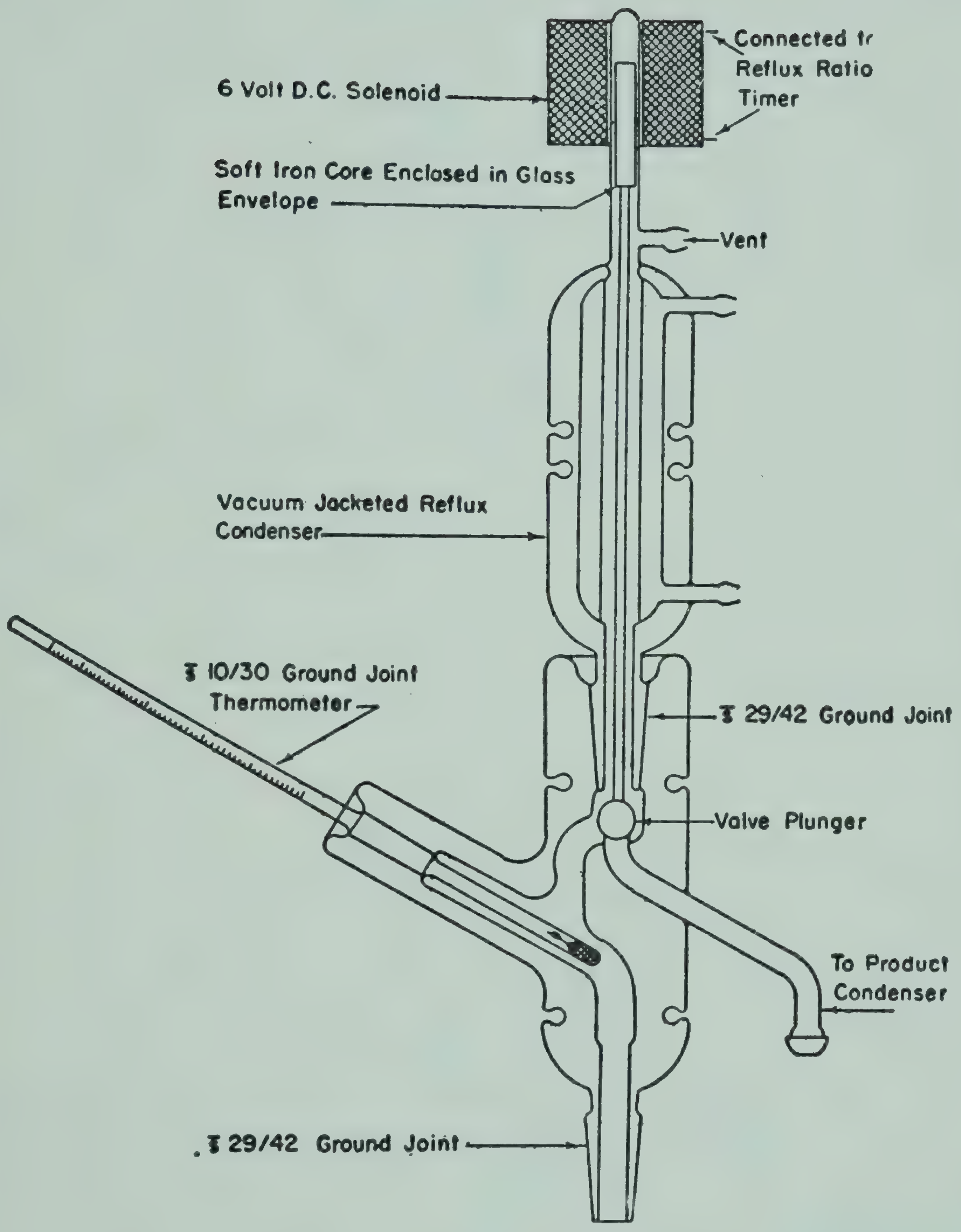

Connected to Reflux Ratio Timer
6 Volt D.C. Solenoid
Soft Iron Core Enclosed in Glass Envelope
Vent
Vacuum Jacketed Reflux Condenser
Ŧ 10/30 Ground Joint Thermometer
Ŧ 29/42 Ground Joint
Valve Plunger
To Product Condenser
Ŧ 29/42 Ground Joint

Figure 39. Automatic vapor dividing still head. (From A. L. Glasebrook and F. E. Williams in *Technique of Organic Chemistry*, Vol. 4, Interscience, New York, 1951.)

Figure 40 shows a still head with a separator on the take-off line. When a mixture is being distilled that separates into two phases upon condensing, this separator will conveniently allow either the upper or lower phase to be taken off, while returning the other phase to the column as reflux.

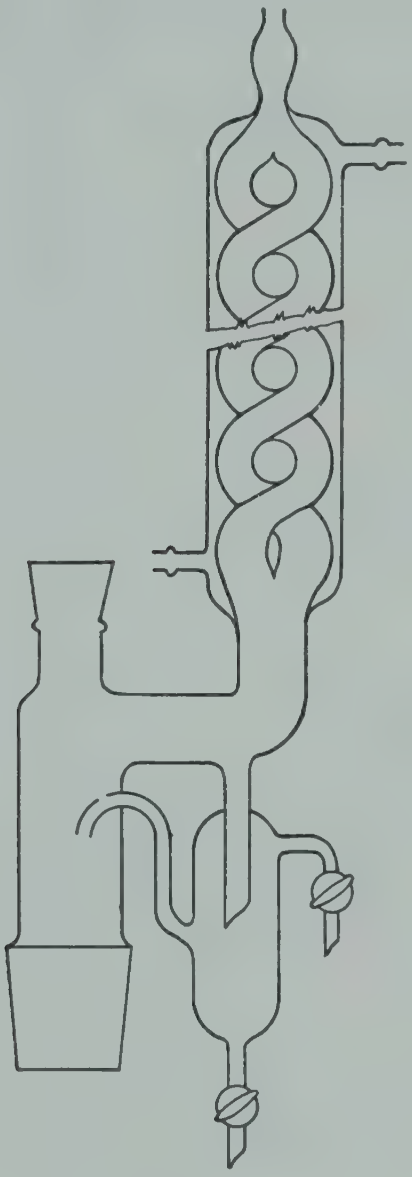


Figure 40. Still head with separator.

Boiling Kettles

One of the most widely used boiling kettles is a standard round-bottomed flask which has a thermowell and which is heated by means of a Glass-Col heating mantle. A type of boiling flask which can be prepared in the worker's laboratory is shown in Figure 41A. This flask is economical to operate and very sensitive to the heat input. The same immersion heater may be used for several flasks but different heaters must be made for various

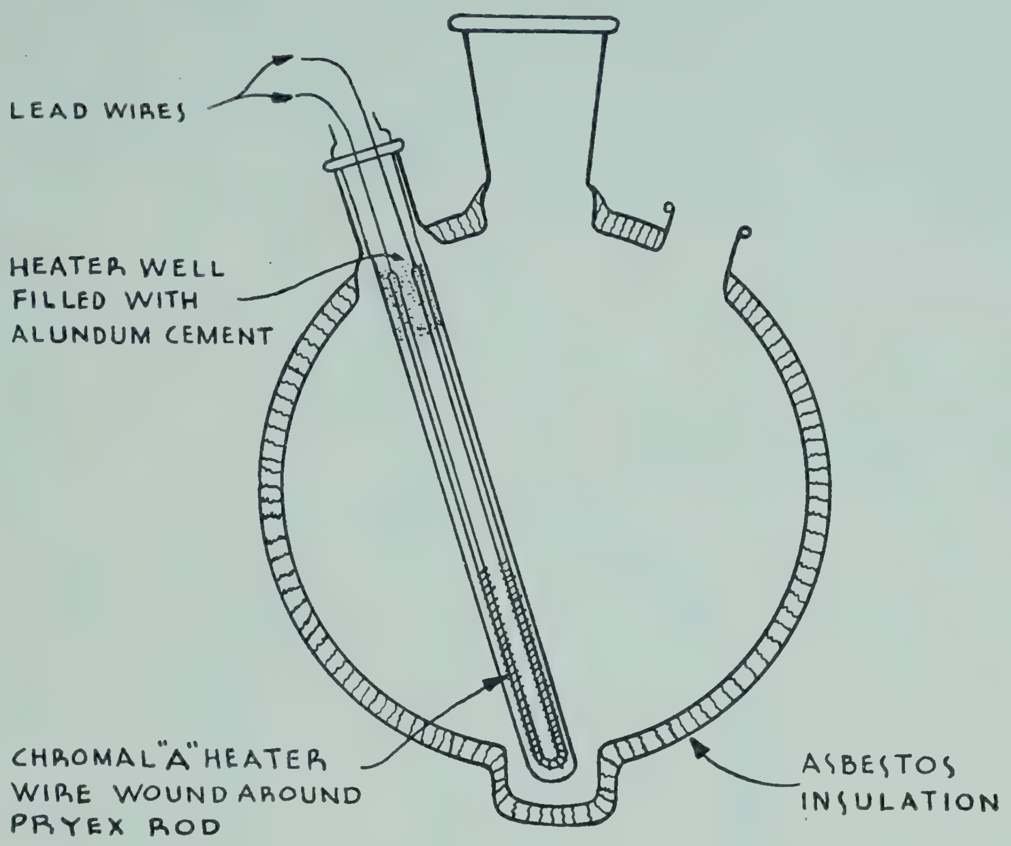

LEAD WIRES
HEATER WELL FILLED WITH ALUNDUM CEMENT
CHROMAL "A" HEATER WIRE WOUND AROUND PRYEX ROD
ASBESTOS INSULATION

Figure 41A. Boiling flask with immersion heater.

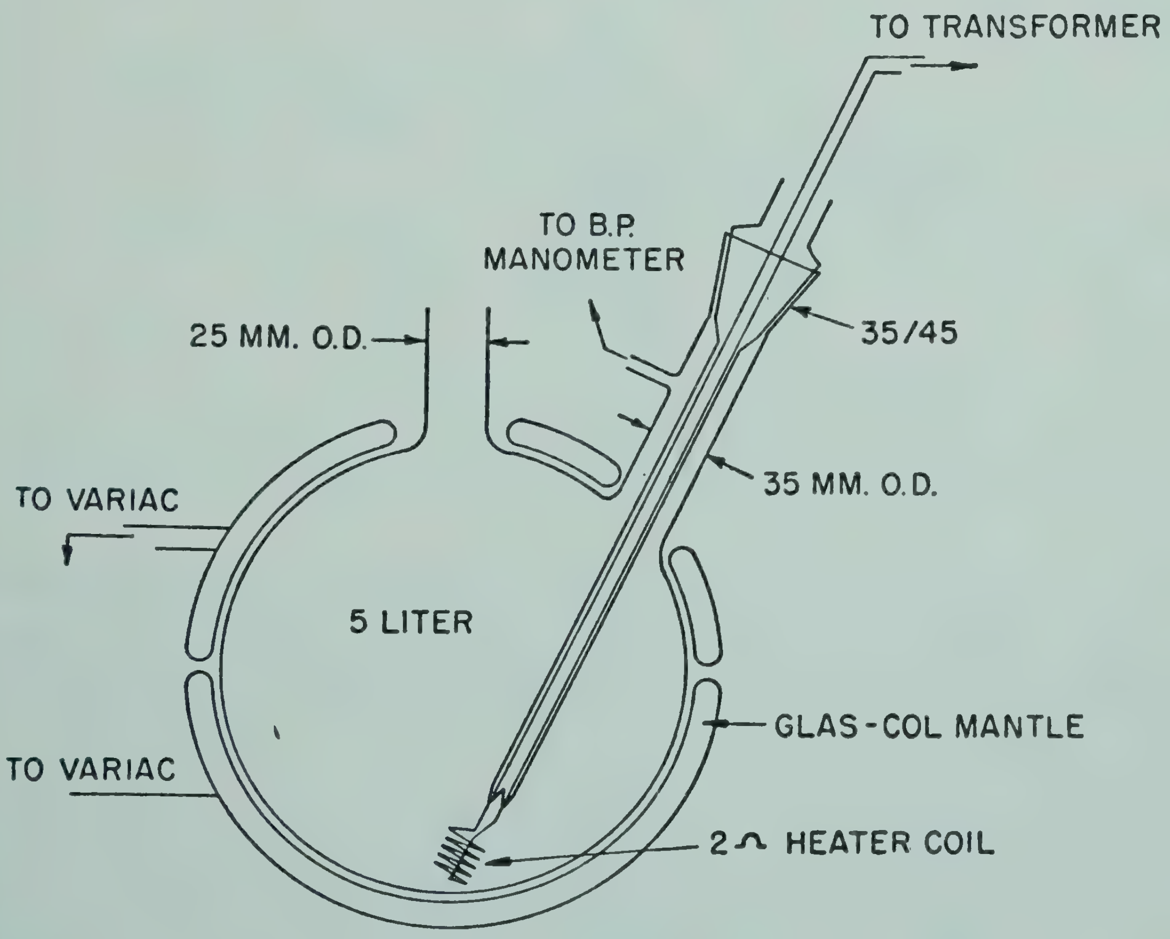

TO TRANSFORMER
TO B.P. MANOMETER
25 MM. O.D.
35/45
35 MM. O.D.
TO VARIAC
5 LITER
GLAS-COL MANTLE
TO VARIAC
2 Ω HEATER COIL

Figure 41B. Boiling flask with Glass-Col heating mantle and immersion heater. (From A. L. Glasebrook and F. E. Williams in *Technique of Organic Chemistry*, Vol. 4, Interscience, New York, 1951.)

sizes of flasks. In Figure 41B is shown a boiling flask with both a Glass-Col heating mantle and an immersion heater.

Boiling flasks with inverted thimbles for coil heaters may be made as shown in Figure 42.

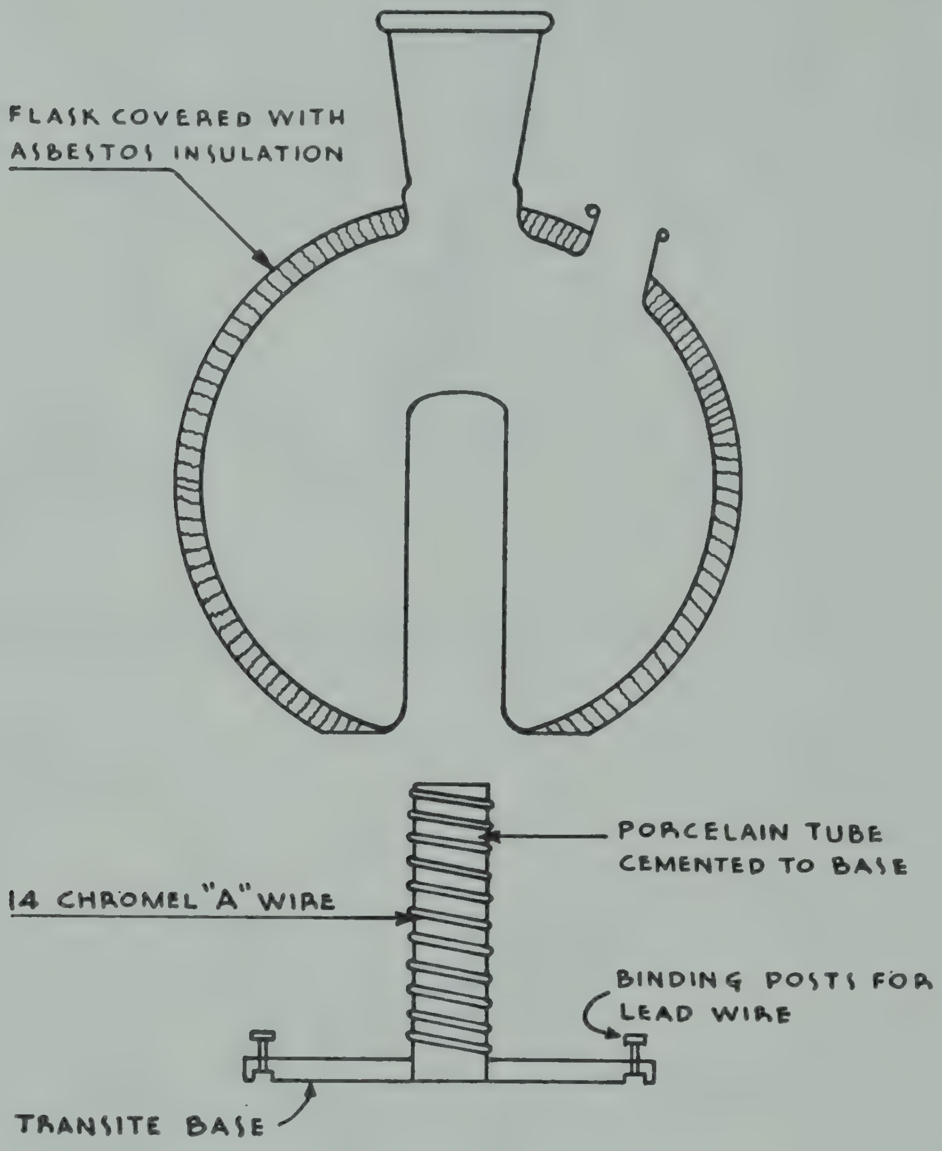

FLASK COVERED WITH
ASBESTOS INSULATION
PORCELAIN TUBE
CEMENTED TO BASE
14 CHROMEL "A" WIRE
BINDING POSTS FOR
LEAD WIRE
TRANSITE BASE

Figure 42. Boiling flask with inverted thimble for coil heater.

Low-temperature distillations require vacuum-jacketed boiling flasks. These can be made as shown in Figure 43. Vacuum-jacketed flasks may be heated either by immersion heaters or inverted thimble heaters.

An excellent method of heating small boiling flasks is shown in Figure 44. The flask is protected from stray drafts of cool air by a vacuum Dewar and heating is by means of a piece of Corning Glass Works[54] E.C. coated plate glass.

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[54] Corning Glass Works, Corning, New York.

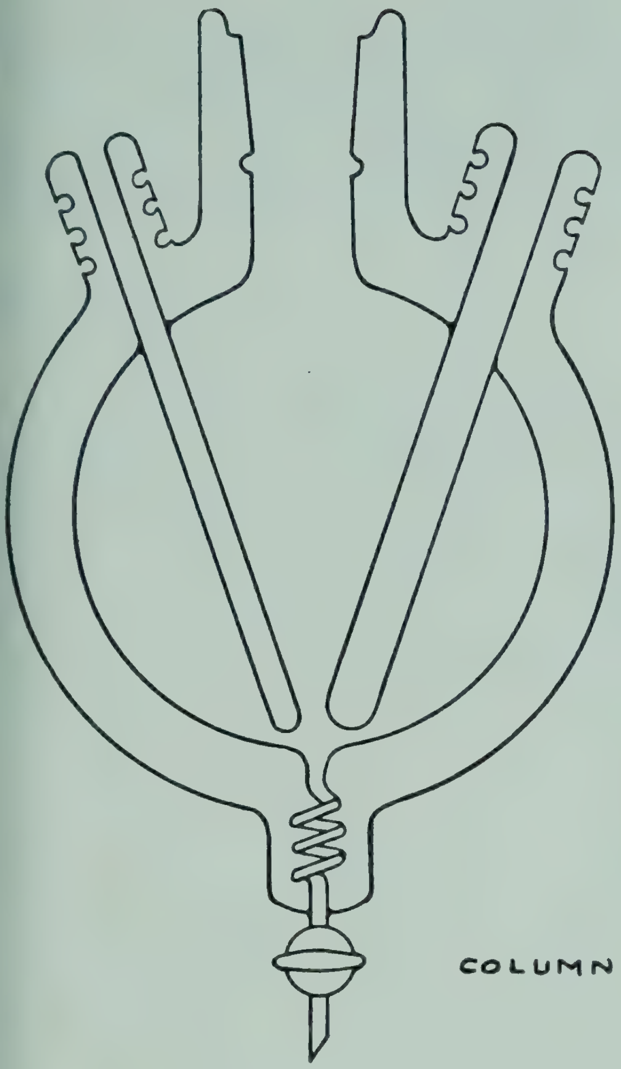

Figure 43. Vacuum-jacketed boiling flask.

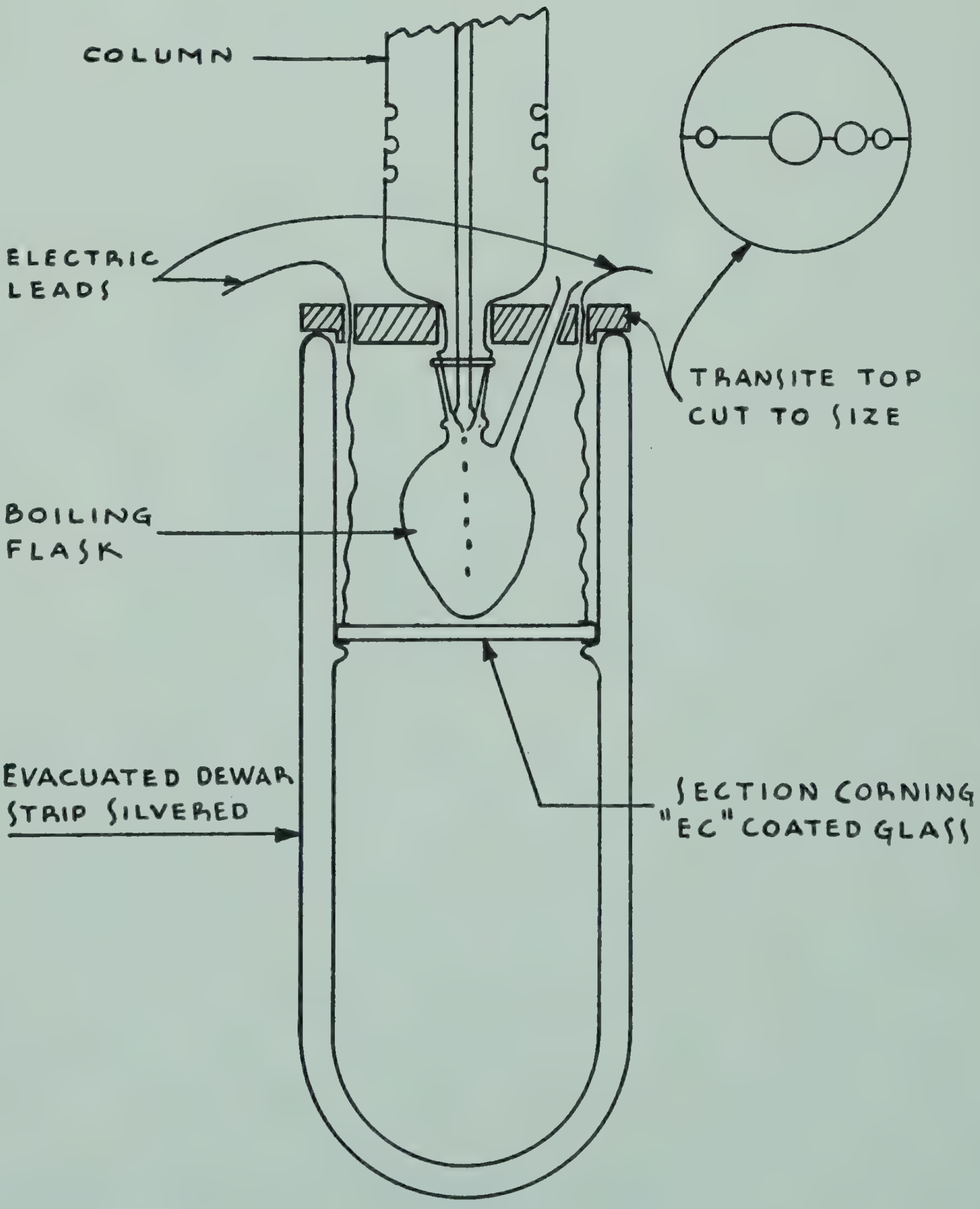
COLUMN
ELECTRIC LEADS
TRANSITE TOP CUT TO SIZE
BOILING FLASK
EVACUATED DEWAR STRIP SILVERED
SECTION CORNING "EC" COATED GLASS

Figure 44. Heating Dewar for micro columns.

The Dewar is strip silvered for observing the sample in the flask. A Transite cover is made having four holes drilled along the center line: one large hole in the center to allow entry of the column into the Dewar, a second hole for inserting a thermometer

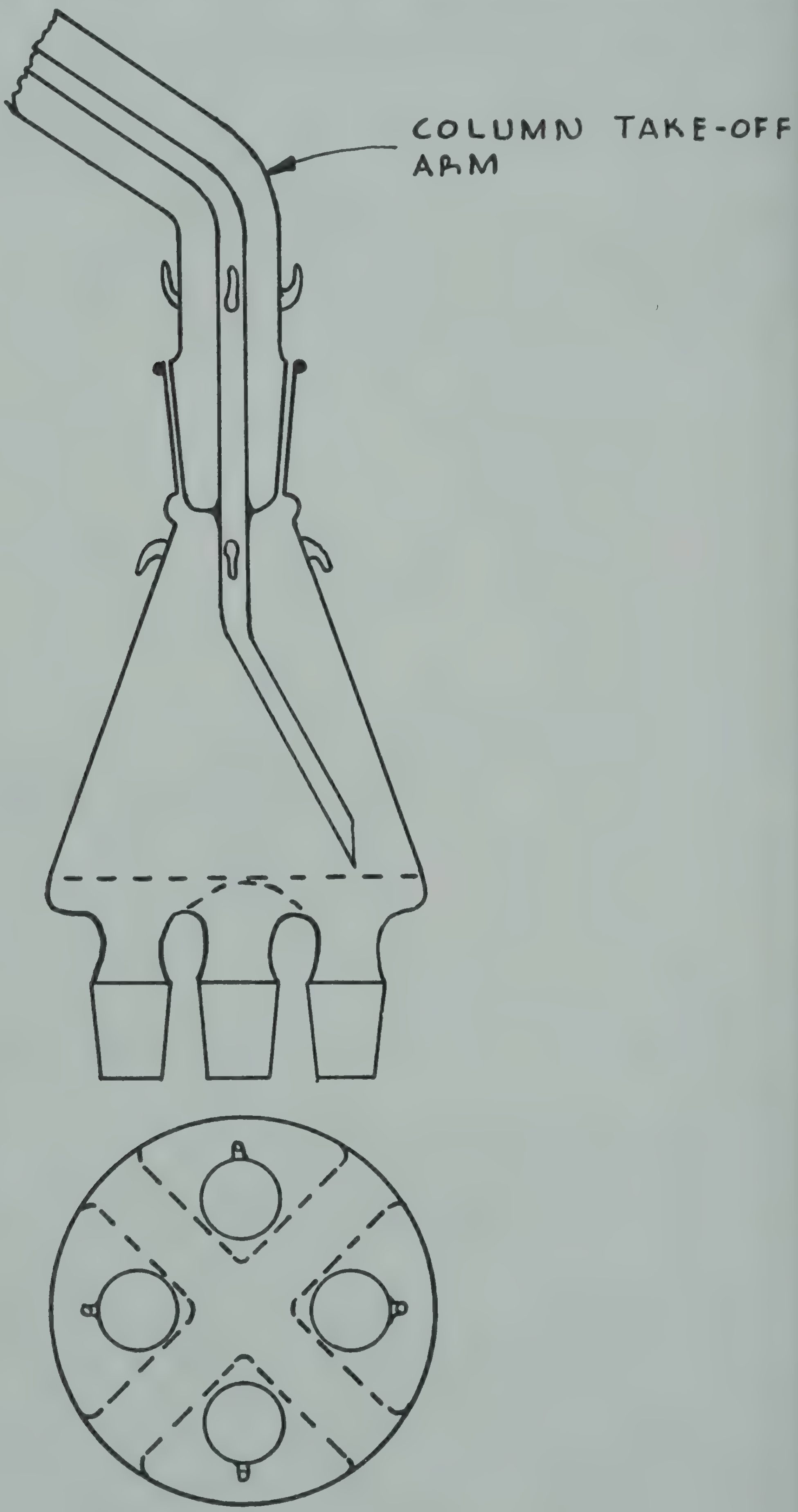

Figure 45. Udder-type fraction cutter.

in the thermowell of the flask, and two small holes for the electrical wire to the heating plate. The Transite cover should then be cut in two along these holes so that the cover may be put in place over the Dewar.

Fraction Cutters

The procedure for making a water-jacketed fraction cutter is described in Chapter VI and shown in Figure 18 (page 128). By means of the vacuum manifold, a full receiver may be opened to the atmosphere during a low-pressure distillation, and a fresh receiver placed in position and evacuated to the same pressure as the column without disturbing the column equilibrium.

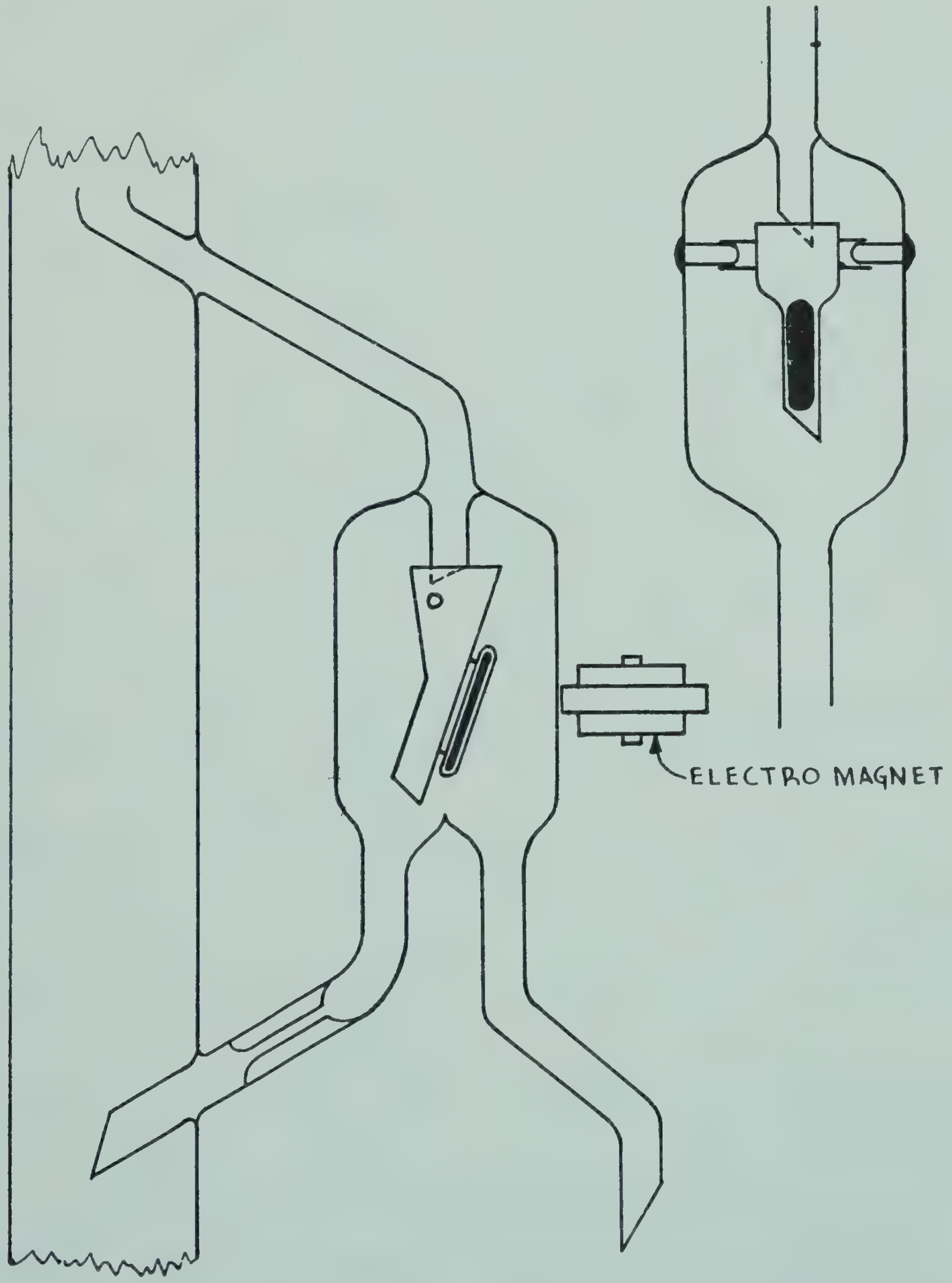

Figure 46. Swinging funnel fraction cutter.

An udder-type fraction cutter is generally made with four, six, or eight outlets, and only that number of separate fractions may be removed without disturbing the column equilibrium. (See Figure 45.)

A swinging funnel type of fraction cutter is shown in Figure 46. This may be sealed to the side of a column as illustrated, or built into the head.

A fraction cutter which eliminates all possible contamination of the distillate by stopcock grease is shown in Figure 47. Collector tubes are inserted into the receiver through the bottom 24/40 $ joints. During reduced-pressure distillation the central stopcock arrangement provides for the opening of either arm of the cutter,

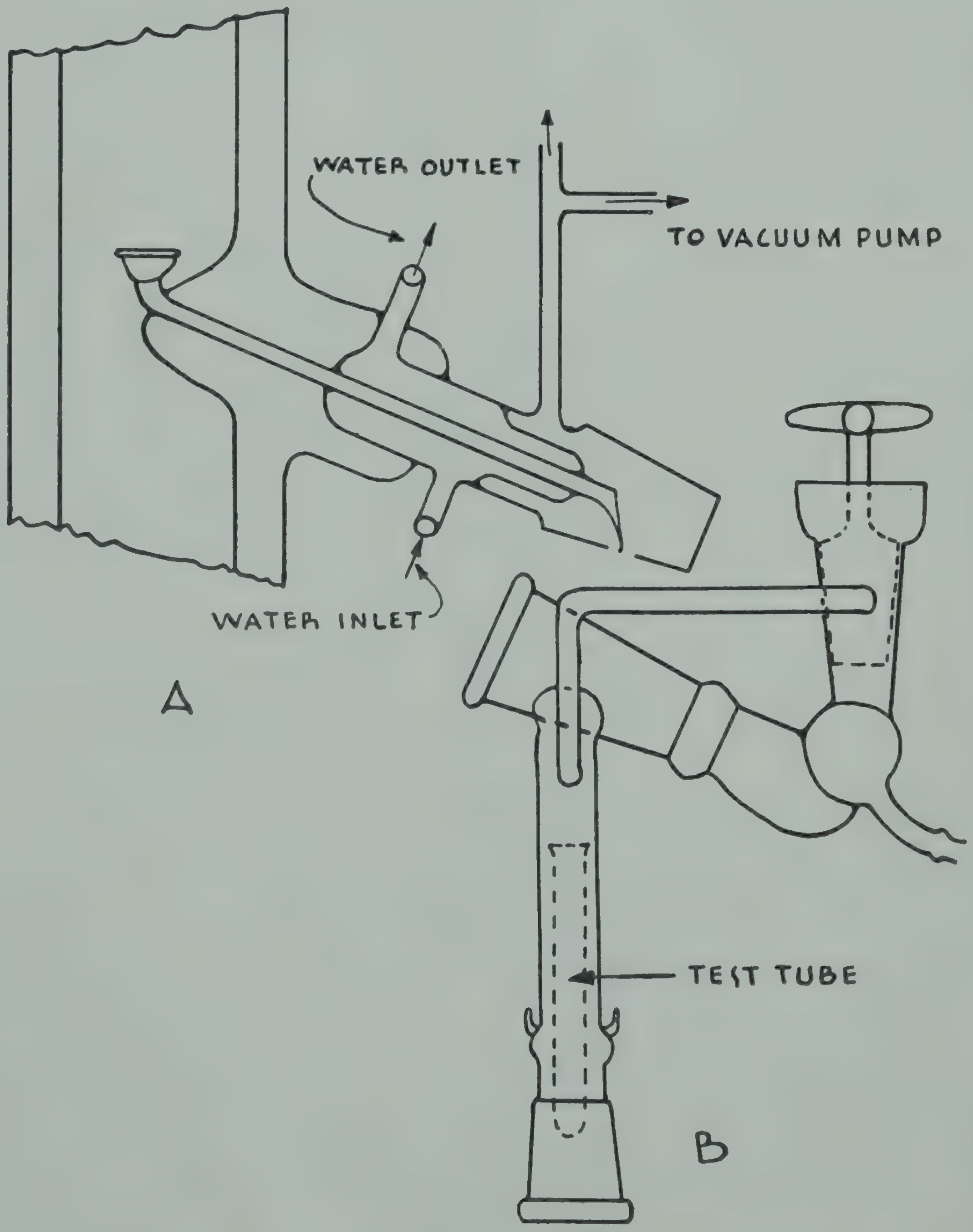

Figure 47. Grease-free fraction cutter.

replacing a full collector tube, and re-evacuating the arm to the same pressure as the other arm and the column without disturbing the column equilibrium. The arm in use should be immersed in a dry ice bath, and an equalizing line is sealed inside the arm and nearly to the bottom to prevent any vapors from being drawn into the vacuum pump.

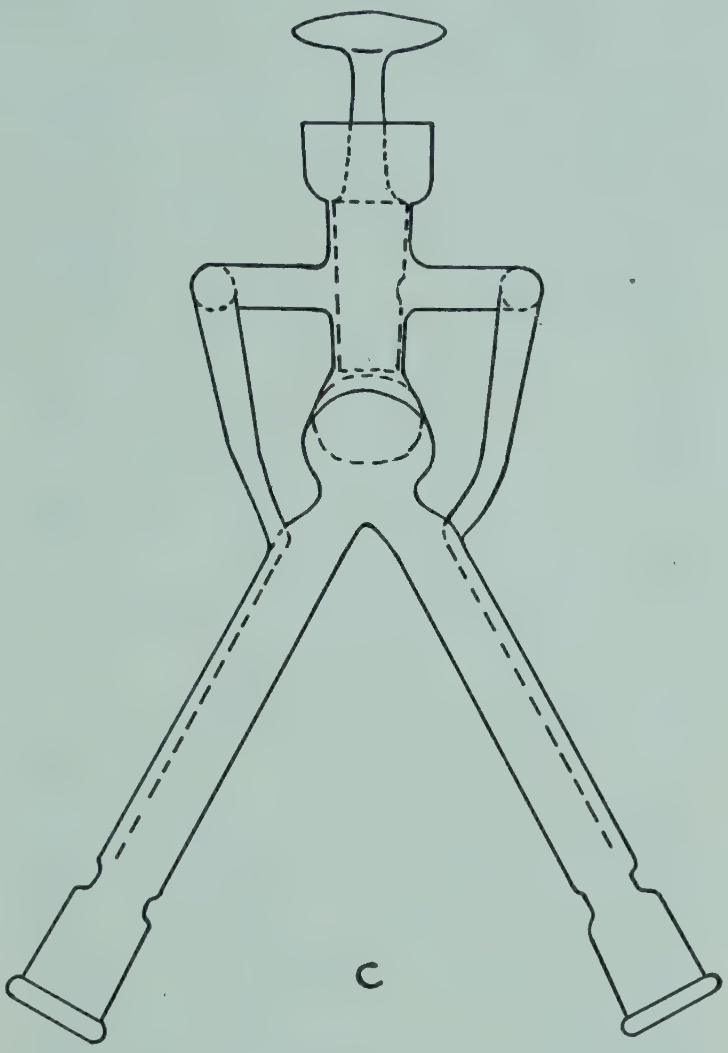

Figure 47. (Continued.)

During the distillation, distillate flowing down the take-off arm passes through a condenser, then through a hole in the $ joints, directly into the collector tube from the drip tip.

CARE AND USE OF A REFRACTOMETER

The extensive use of refractometers in evaluating the efficiency of the separation accomplished by fractional distillation prompts these notes on the care and use of a refractometer.

Description

Figure 48 is a photograph of the Abbe 56 refractometer manufactured by the Bausch and Lomb Optical Company.[55]

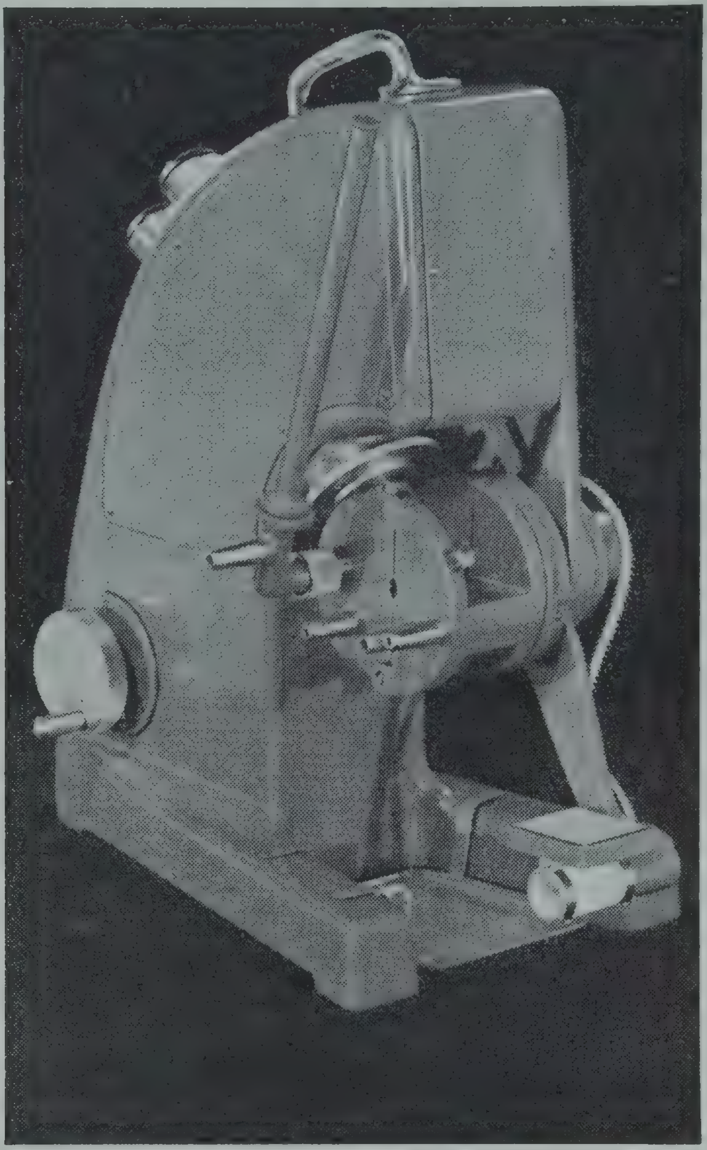

Figure 48. Abbe refractometer.

This is a precision instrument and its continuous good performance will be maintained only if the operator is familiar with the instrument and is willing to give it the proper care. The various parts of the refractometer, shown in Figures 49 and 50, are as follows:

1. Refracting prism case
2. Illuminating prism case
3. Prism case pin
4. Prism case locking spring
5. Filling tabulation
6. On-off switch for field lamp
7. Hold-down switch for scale lamp

[55] Rochester 2, New York.

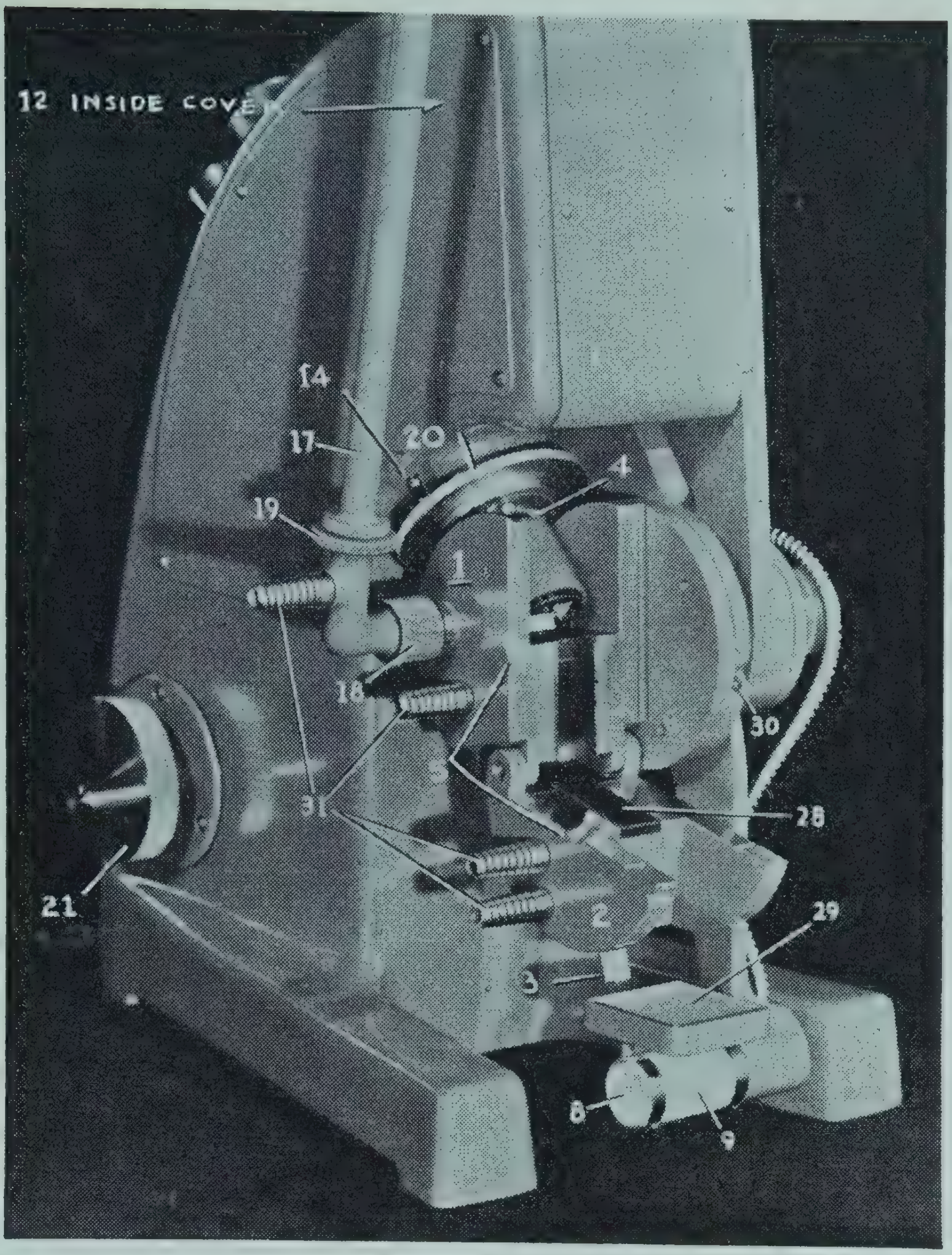

Figure 49. Location of parts in Abbe refractometer.

8. Field lamp
9. Plastic diffuser
10. Rotating lamp arm
11. Scale lamp
12. Scale plate
13. Scale eyepiece
14. Compensator dial locking screw
15. Ground-glass window
16. Field eyepiece
17. Thermometer
18. Thermometer connecting collar
19. Thermometer locking collar
20. Compensator dial
21. Handwheel
22. Driving screw
23. Traveling nut
24. Adjusting screw
25. Screw position No. 1
26. Screw position No. 2
27. Exit pupil
28. Illuminating prism
29. Removable mirror
30. Adjusting lug
31. Nipples for hose adjustments

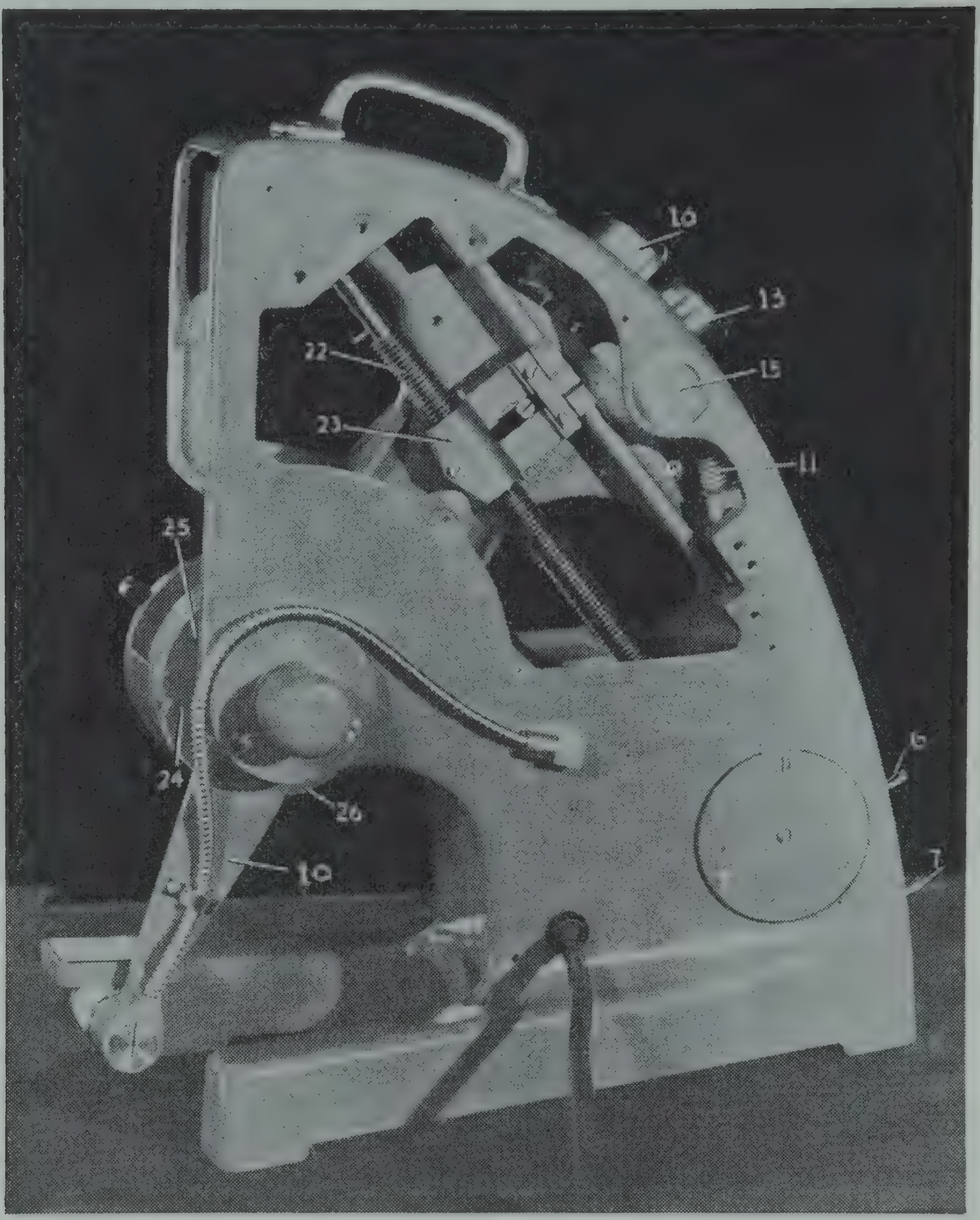

Figure 50. Location of parts in Abbe refractometer.

Any refractometer of the Abbe type consists of a refracting prism system with an appropriate scale and a compensation system consisting of Amici prisms which permit the use of white light. A telescope with cross hairs permits the border line of total reflectivity to be observed and set accurately.

Liquids are measured by introducing a thin film of the liquid between the upper and lower refracting prisms, and solids are measured by affixing them to the surface of the upper prism by means of a suitable contact liquid. The contact liquid must have a higher index than the sample to be measured.

The Prism System. The hinged prism case (2, in Figure 49) carrying the illuminated prism is opened by a slight forward pressure on the pin (3). The prism housings are hollow to provide for a flow of liquid to hold the prism system at a constant temperature.

The Scales. The index and "total solids" scales are on a transparent glass plate (12) which is attached to the sector arm inside the housing and are read through the lower eyepiece (13). This eyepiece is made with a sliding fit and should be focused to give the clearest image of the scale. The scales are designed to read directly to the third decimal place with estimation to the fourth. The "total solids" scale reads directly to 0.5 % with easy estimation to 0.1 %. The adjustment of the scale plate on the sector arm is extremely critical and should not be attempted by anyone not thoroughly familiar with such adjustments.

The Scale-Prism Motion. The handwheel (21) provides the only means of moving the combined scale-prism system. Rotation of the handwheel is transmitted by bevel gears and a screw to the traveling nut on the sector. Any slight backlash which may exist in this system has no adverse effect on the accuracy of the setting, since the scales and the prism constitute a rigid member and, hence, always remain in the same alignment.

Compensator System. In the newer Abbe instrument both prisms of the compensator system move together in the same direction as a unit and the scale dial serves to rotate them. When the correct compensation has been secured, the border line will be achromatic at the center of the field, with a faint red dispersion showing at one extremity and a faint blue dispersion at the other. The system should be set so that the short achromatic section of the border line is centered on the cross hairs.

Illumination. The field lamp (8) is carried in a plastic diffusing chamber at the extremity of the swinging arm. This lamp is controlled by a switch (6). A second toggle switch (7), which is a hold-down type, controls a light for illuminating the scale field. If daylight illumination is desired, a small mirror may be clamped over the plastic cylinder (29) and rotated into the best position for illumination.

Thermometer. The thermometer (17) is attached in an upright position by means of a knurled collar (18). By loosening this collar slightly the thermometer may be adjusted to any desired reading position.

Drip Pan. A small drip pan is provided to catch any liquid which may escape from the prism system during filling.

Care of the Instrument

The Abbe refractometer was designed to provide maximum convenience and accuracy of reading, and to protect all parts as far as possible from the sample materials and solvents. In even the best of instruments, however, trouble will ultimately result unless the operator follows certain basic practices in the daily use of the instrument.

Keeping Refractometer Clean. The refractometer must be kept scrupulously clean at all times. Dust, oil, and solid materials will eventually find their way into the bearings and hinges if allowed to accumulate on any part of the instrument. This will cause wear and eventual misalignment. To avoid this, the operator should make it a practice to clean all exposed surfaces thoroughly when he is through with the instrument or, during continual use, it should be cleaned at the end of each day's work.

Keeping Prism Clean. The prisms should be thoroughly cleaned after each test and should be kept closed when not in use. The glass from which the prism is made is of high refractive index and inherently soft. Therefore, it is easily damaged by surface scratching and corrosion. Dust, if allowed to settle on the polished surfaces, may cause more damage during its removal than many hours of actual service. Any deterioration of the surface quality results in a hazy border line; hence, every possible precaution should be taken to protect and preserve the prism surfaces. Special care should be taken when solid materials are being measured. Bausch and Lomb recommend the following procedure for cleaning the prism surfaces.

1. A cotton swab is saturated with a suitable solvent (benzene for organic materials and distilled water for water-soluble materials), and the swab is passed lightly over the surfaces until they show no tendency to streak.

2. This process is repeated with petroleum ether until bot

the glass and adjoining metal surfaces are clean. The surfaces should not be dried by rubbing with cotton.

3. Lens tissue, if kept in a closed container, may be employed if used lightly. *Caution:* The use of any cleaning material which has been lying on the work table, where it can pick up dust and grit, should be avoided.

4. External exposed glass surfaces of lenses and prisms may be safely cleaned with a cleaning tissue lightly moistened with benzene. The scale plate and reticule should be cleaned with a camel hair brush. Solvents should not be used.

Oiling. The bearings of the screw (22) together with the bevel gears and traveling nut (23) should be given a drop of light machine oil occasionally. This also applies to the prism case hinge.

Use of the Standard Test Piece. Each instrument is provided with a standard test piece for testing and adjusting the scale and compensator position. For complete instructions on this operation, the reference manual supplied by Bausch and Lomb should be consulted.

Method of Reading

The following general directions apply in making any reading with the instrument.

A. After the sample is in position on the instrument, the scale is set at the approximate value expected using the scale eyepiece.

B. The border line, which will probably be strongly colored, is brought near the cross hair and the color compensated for by adjusting the position of the dial (20). The border line should be faintly blue at one end and faintly red at the other.

C. The eyepiece (16) is focused sharply on the cross hairs and the dividing line brought upon their intersection by means of the handwheel.

D. The index is read through the scale eyepiece (13), and the fourth place estimated at the time of reading.

Work Involving Transparent and Viscous Liquids

For measurements of liquids and especially those of organic nature, hydrocarbons, vegetable oils, and the like, accurate temperature control and thorough cleaning of the prisms between samples are necessary. If the liquid is free flowing or only slightly viscous, it may be introduced through the tubulation (5) by means of a pipet or dropper. Very viscous materials are best placed upon the lower illuminating prism and spread over the face of the prism with a wooden applicator. Caution: Glass or metal applicators should never be used against the face of the prism.

In order to obtain the best reading with any liquid or semiliquid, it is essential that the space between the two prisms be uniformly filled. Nonuniformity of filling will result in dark areas in the bright disc. If the sample is contaminated through improper cleaning, darker areas or striation may extend into the bright area. When this occurs the prism should be thoroughly cleaned and a new sample inserted.

Bubbles created in the sample during insertion between the prisms causes darkened areas which may sometimes be eliminated by opening and closing the prisms slightly two or three times. Bubbles are most often caused by the adherence of foreign materials, especially those of an oily nature, to the glass surfaces as the sample flows in from the tubulation. This emphasizes the need for thorough cleaning of the prism surfaces between samples.

Temperature Control

Materials differ greatly in their change of index with respect to a change in temperature. How closely the temperature of the sample must be controlled depends entirely upon the nature of the material and the accuracy of the reading desired.

The list below gives several materials together with the change of index of refraction per degree centigrade.

Water	0.0001
Linseed oil	0.0004
Carbon disulfide	0.0008
Crown glass	0.00002
Flint glass	0.00001
Fluorite	0.00001

From this list it can readily be seen that with some liquids a change of 1 degree will cause errors extending well into the fourth place. Since change of 1 degree will cause a change of 0.00001 for water solutions, a temperature control of approximately 0.5° C. will be necessary to secure correct readings. For carbon disulfide the 1-degree change will cause an 0.0008 change in index; thus the temperature should be controlled within approximately 0.05° C. for correct readings. These two cases represent extremes, but vegetable or animal oils and fats and the hydrocarbon series have coefficients of the order of 0.0004 and 0.0005 per degree, and hence the temperature must be well controlled if accurate results are to be obtained. A precision temperature control unit is manufactured by the Precision Scientific Co., Chicago, Illinois.

XIII

HIGH VACUUM

The design and construction of vacuum equipment for research work, as well as the widespread use of rare-gas discharge tubes and other evacuated apparatus which a glassblower must make, entails a knowledge of the principles of high vacuum. High-vacuum theory and practices have been well covered by such authors as Dushman,[1] Martin and Hill,[2] and Kaye.[3] A description of all the methods or design of apparatus for pumping a vacuum would in itself be a complete book. The technique discussed in the following pages, as well as the equipment described, will enable a glassblower to meet many of the situations involving high vacuum which ordinarily arise in research laboratories.

HISTORY

The science of the production and measurement of high vacuum extends over 300 years. In 1643 Galileo's assistant Torricelli discovered the possibility of producing vacuum above a column of mercury by filling a 30-in. closed end tube with mercury and inverting it in a reservoir of mercury. In 1650 von Guericke devised a mechanical air pump with which he obtained pressures as low as 1/1000 of an atmosphere. Von Guericke had become famous by 1664 for his experiment with the Magdeburg hemispheres, which, when evacuated, could not be pulled apart by two teams of eight horses each. In 1865 Toepler developed the pump named after him and still in use in modified form. Today,

[1] Dushman, Saul, *Scientific Foundations of Vacuum Technique.* Wiley, New York, 1949.

[2] Martin, L. H., and Hill, R. D., *A Manual of Vacuum Practice.* Melbourne Press, 1946.

[3] Kaye, G. W. C., *High Vacua.* Longmans Green, London, 1927.

however, the slow speed of these pumps limits their use to special purposes.

It is very probable that the beauty of the electrical discharge in evacuated tubes was responsible for much of the interest shown by the early scientists in obtaining a better vacuum. In Germany in 1838 Geissler originated the discharge tube as we know it today. His tube was made by sealing platinum wires into the glass. However, as early as 1785 another scientist named Morgan had noticed that the color of the discharge afforded a guide to the pressure.

The development of the mercury diffusion pump by Goede in 1915 was a major step in the advance of high-vacuum technique and possibly influenced Langmuir to design his mercury condensation pump in 1916. With these two mercury pumps, pressures as low as 10^{-4} micron were readily obtainable, and it was this factor which made possible the rapid expansion of the radio and electronic industries of that period.

HIGH-VACUUM SYSTEMS

In the production of high vacuum today, vacuum systems are classified as either static or kinetic systems.

Kinetic Vacuum Systems

Kinetic systems feature continuous pumping by very fast pumps against an intentional leak as in a mass spectrometer. Pressures in a kinetic system are rarely lower than 1×10^{-6} mm. of mercury.

Static Vacuum Systems

Static systems, on the other hand, are tight vacuum systems, usually having pumps with slow pumping speeds but producing vacuums of low pressure. Pressures as low as 1×10^{-9} mm. of mercury have been recorded. In static systems all the leaks have been removed and the system is as completely degassed as possible. In addition traps of liquid air or dry ice and a suitable solvent are used to trap stray vapors. A typical example of static systems is one used for the evacuation of fractional distillation column jackets.

A Simple Static System

The vacuum system shown in Figure 1 includes all the essential components of a simple vacuum system. The speed at which the vessel is evacuated is determined not only by the pumps but by the connecting tubes. Kaye[3] states, "The diameter of 2 cm. should be regarded as practically the minimum for connecting tubing for high speed pumping." In high-vacuum systems the pressure is usually so low that collisions between gas molecules occur less frequently than collisions between the gas molecules and the confining walls. Knudsen[4] found that at pressures of less than 10^{-3} mm. of mercury gas flow is independent of the pressure and wall friction alone affected the pumping speed. For air at 0° C. the mean free path is about 7 mm. at a pressure of 10^{-2} mm. For a pressure of 10^{-3} mm. the mean free path is about 7 cm., at a pressure of 10^{-4} mm. it is 70 cm., and for a pressure of 10^{-8} mm. this mean free path has grown to about 7 km. (5 miles). From this the importance of maintaining all connecting tubes as large as possible can be readily seen. In addition it is

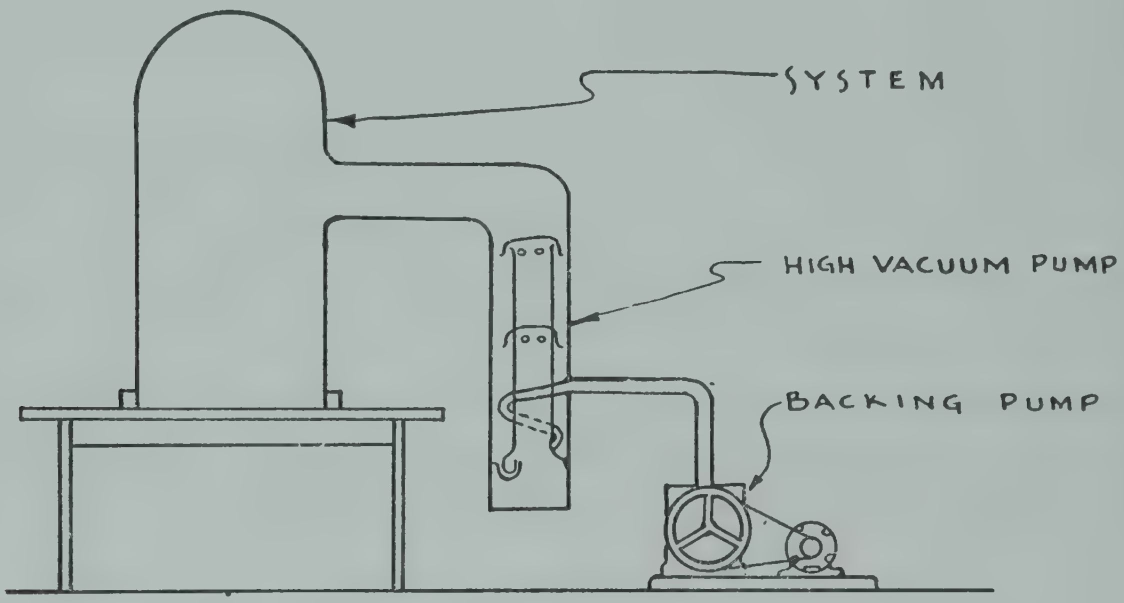

Figure 1. Simple static vacuum system.

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[4] Knudsen, M., *Ann. Physik*, **28**, 76 (1909).

desirable to keep the connecting tubes free from constrictions and sharp bends in order to reduce the wall friction.

VACUUM EQUIPMENT

Backing Pumps

One of the first requisites for a vacuum system is a good backing pump. Among those obtainable commercially are the Welch Duo-Seal,[5] one- and two-stage pumps. These pumps are guaranteed to pump a vacuum 0.05 micron and they have a pumping speed of 33.4 liters per minute. A two-stage model of this same pump has a pumping speed of 58 liters per minute and a guaranteed vacuum of 0.0001 mm. Hg. The Central Scientific Co.[6] produce a series of three pumps, the Hivac, the Megavac, and the Hypervac. The Hypervac has free air pumping speed of 250 liters per minute and an ultimate vacuum of below 0.1 micron In operation the Welch pump runs more quietly as the vacuum improves, whereas the Cenco pump gives an indication of the pressure by the sharp snapping sound with which the pump operates.

Backing pumps play an essential part in the successful operation of diffusion pumps. They not only carry gas away from the vapor pump but maintain the backing pressure lower than the minimum value above which the vapor pump ceases to operate.

Backing pumps should be well protected by traps on the inlet side of the pump. When a mercury diffusion pump is being used, one of the traps should be immersed in a dry ice mixture, and in addition chemical traps suitable for removing undesirable elements from the gas before it enters the pump should be used. The condition of the oil as well as the oil level of the pump should be watched and the oil changed at reasonable intervals.

Mercury Diffusion Pumps

Modern diffusion pumps are divided into two classes, mercury vapor and oil diffusion pumps. In the first, mercury vapor

[5] W. M. Welch Scientific Co., 1515 Hedgwik Street, Chicago, Illinois.

[6] 1700 Irving Park Road, Chicago 13, Illinois.

is the pumping medium and in the latter the pumping medium is specially refined oil or high-boiling organic liquids.

In the original diffusion pump by Goede (see Figure 2), gas

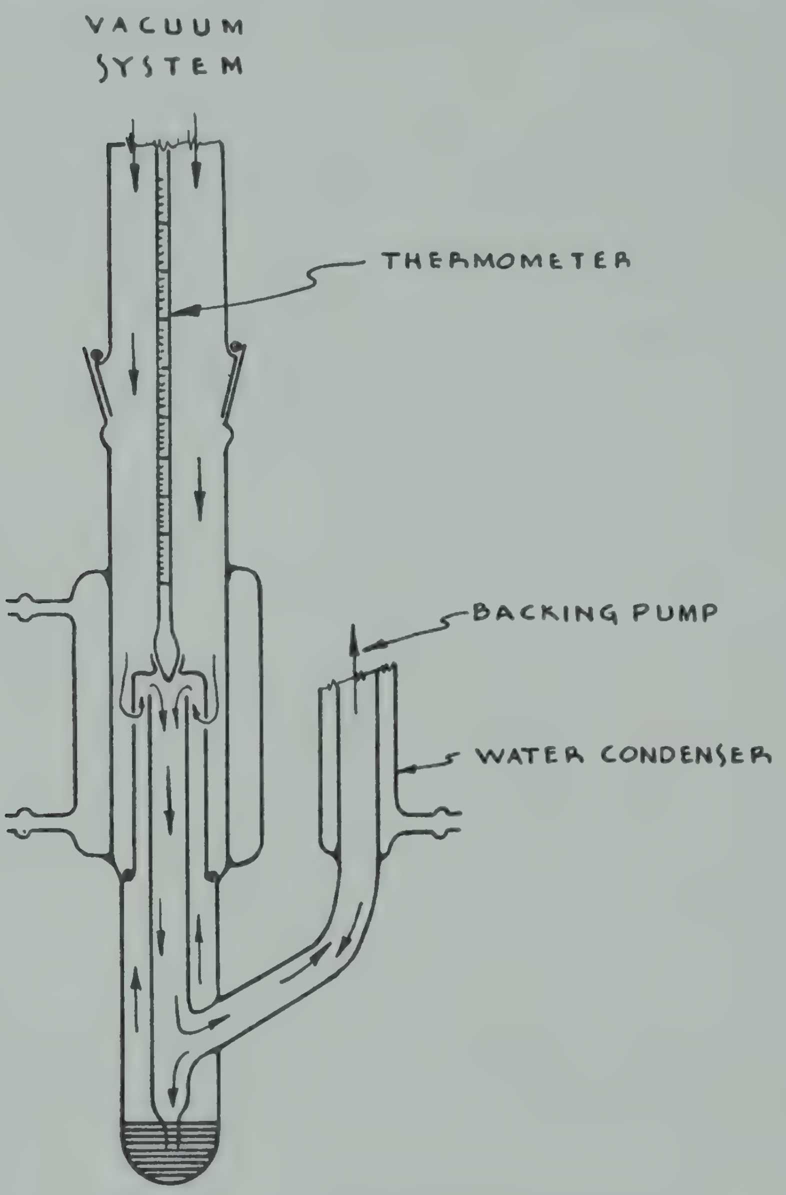

Figure 2. Goede mercury diffusion pump.

from the high-vacuum system diffused through a narrow cylindrical slit into a stream of mercury vapor. There, by collision with the mercury atoms, the molecules of gas were swept along into the section of the pump where the mercury was condensed and the gas pumped off by the backing pump. In this pump there was some diffusion of mercury back into the high-vacuum system. Goede attempted to keep the back diffusion to a minimum by using a narrow slit (0.1 mm.) and by controlling the mercury vapor density. The maximum efficiency in pumping speed is obtained with this pump when it is operated in such a way that the mean free path of the gas molecules in the vapor stream is of the same magnitude as the width of the slot. Langmuir's pump (Figure 3) eliminated the necessity of a narrow slit and opened the way to the high-speed pumps of today.

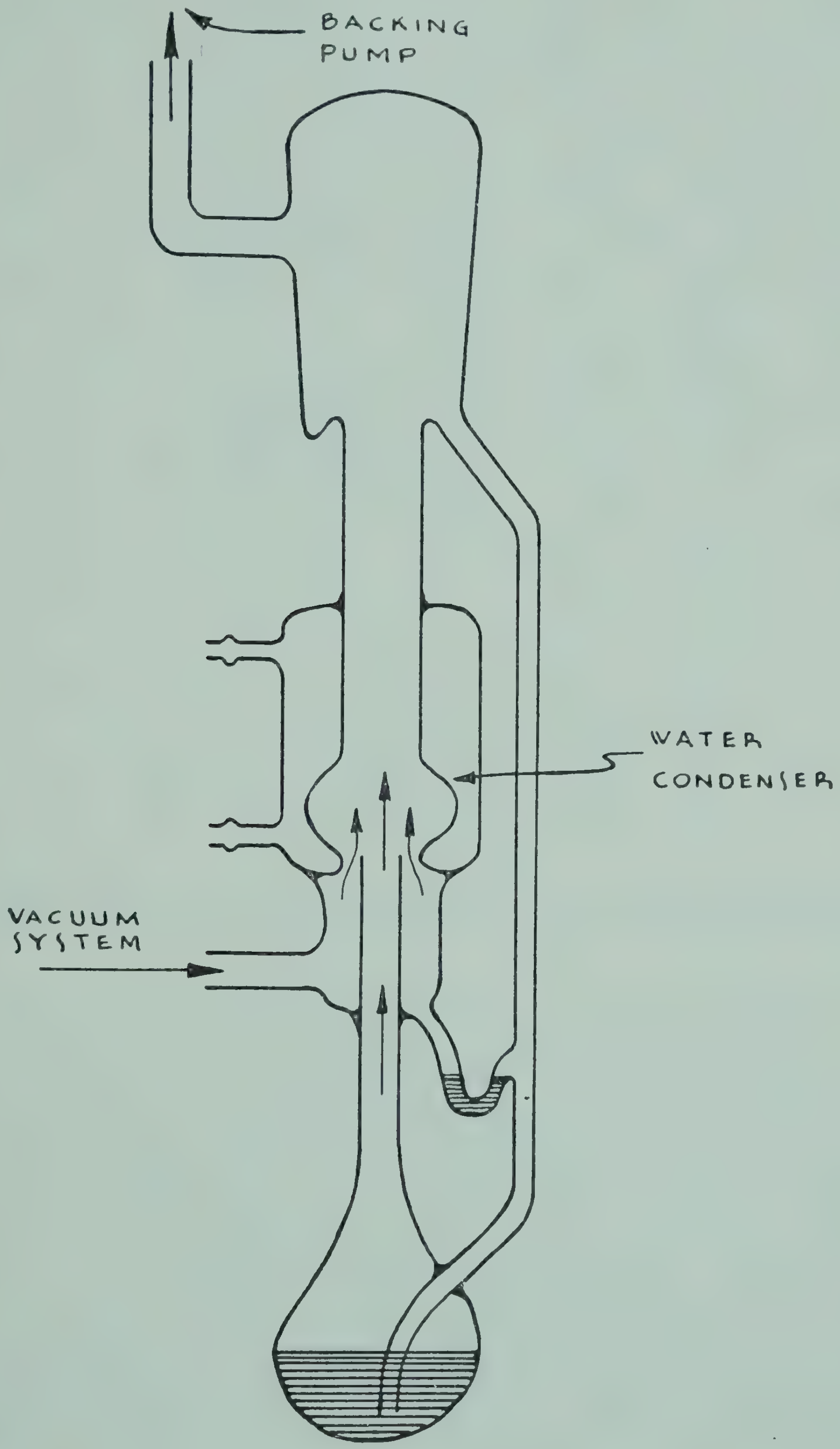
BACKING PUMP
WATER CONDENSER
VACUUM SYSTEM

Figure 3. Langmuir mercury diffusion pump.

Construction Data. In Figure 4A-D is shown a vertical single-stage, umbrella-type mercury diffusion pump. These pumps have a pumping speed of about 6 liters per second and because of their small size are easily adaptable to most installations. The following procedure is recommended in their construction.

The four parts of the pump, A, B, C, and D, should be constructed before actual assembly of the pump is started. The chimney A is a piece of 19 mm. O.D. tubing to which a section of 14 mm. O.D. tubing is sealed. The seal should be pulled into a smooth taper as shown and then cut off, leaving about 20 mm. of the smaller diameter tubing. The umbrella (B) is made from 24 mm. O.D. tubing and held in position on the chimney with a small wrapping of copper screening. To facilitate blowing into the umbrella while sealing it to the chimney supports, dampened asbestos paper may be wedged into the slit formed between the chimney and umbrella. This asbestos should be removed before inserting the chimney and umbrella in the condenser section. The condenser (Figure 4C) is made next following the standard procedure for making condensers (see page 105), and the boiler is made following the instructions for making Dewars (see page 84).

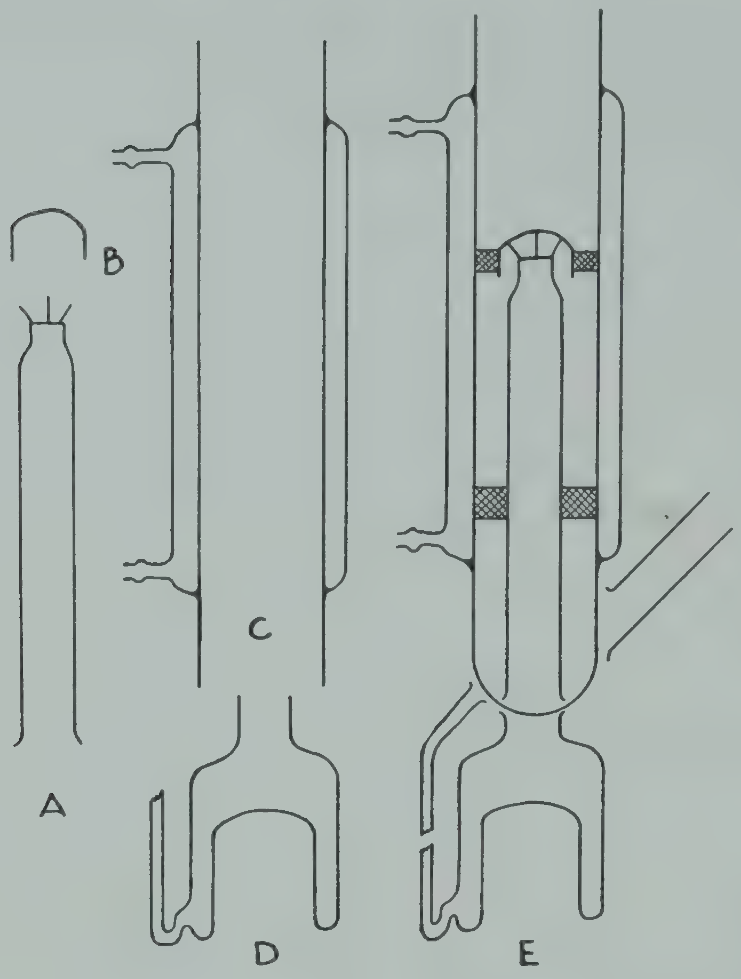

Figure 4A-E. Single-stage mercury diffusion pump.

After the chimney is supported in the condenser as shown in Figure 4E, the lower ring seal may be made and the boiler sealed in place. The high-vacuum side arm and mercury return line connections are then sealed on. In aligning the mercury return line of the boiler with respect to the condenser, a red grease pencil mark placed on each section will be helpful and will make it easier to avoid turning one piece faster than the other while rotating. Some asbestos cloth tape wrapped around the lower

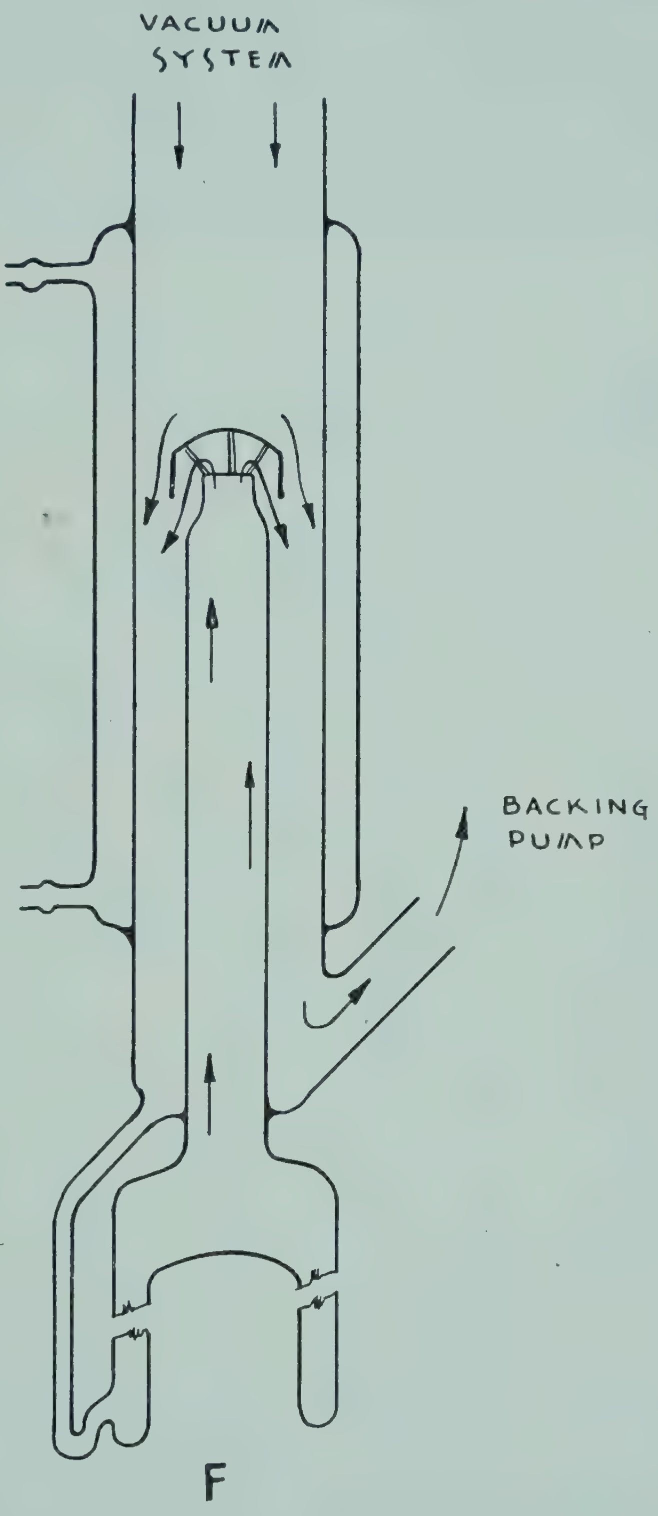

Figure 4F. Completed mercury diffusion pump.

ring seal of the condenser will protect it from direct flame splash while the second ring seal is being made and boiler attached. The pump is finished by sealing in a section of glass to complete the mercury return line to the boiler. It is advisable to make and seal a short (6-in.) condenser to the low-vacuum side of the pump. This should be of the same inside diameter as the side arm itself. The finished pump is shown in Figure 4F. A modification of this single-stage mercury diffusion pump is shown in Figure 5. The difference between the pumps shown in Figures 4 and 5 is in the method of condensing the mercury vapor in the low-vacuum or backing pump side. The coil wound around the inside tube of the main condenser replaces the secondary water condenser of the pump in Figure 4. The coil may be wound and then sealed to the inner jacket, or a straight piece of tubing may be sealed to the inner section of the condenser and wound into a coil later. When the latter method is used, a straight side-arm seal is made to the

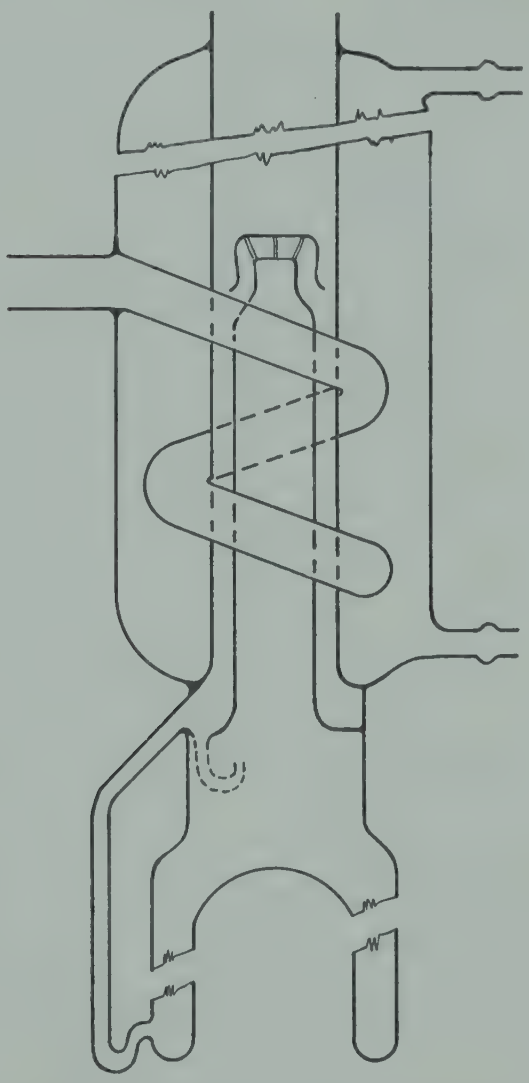

Figure 5. Modified single-stage mercury diffusion pump.

larger tube, and when the seal has cooled sufficiently for the glass to set, a right-angle bend is made close to the surface of the large tube. By heating and bending one small section of tubing at a time in the cross fires, the coil is wound to the desired length. This coil makes the pump more compact and less susceptible to breakage. A second modification that also makes the pump less liable to breakage is to replace the outside mercury return line with a mercury trap inside the boiler, shown with dotted lines.

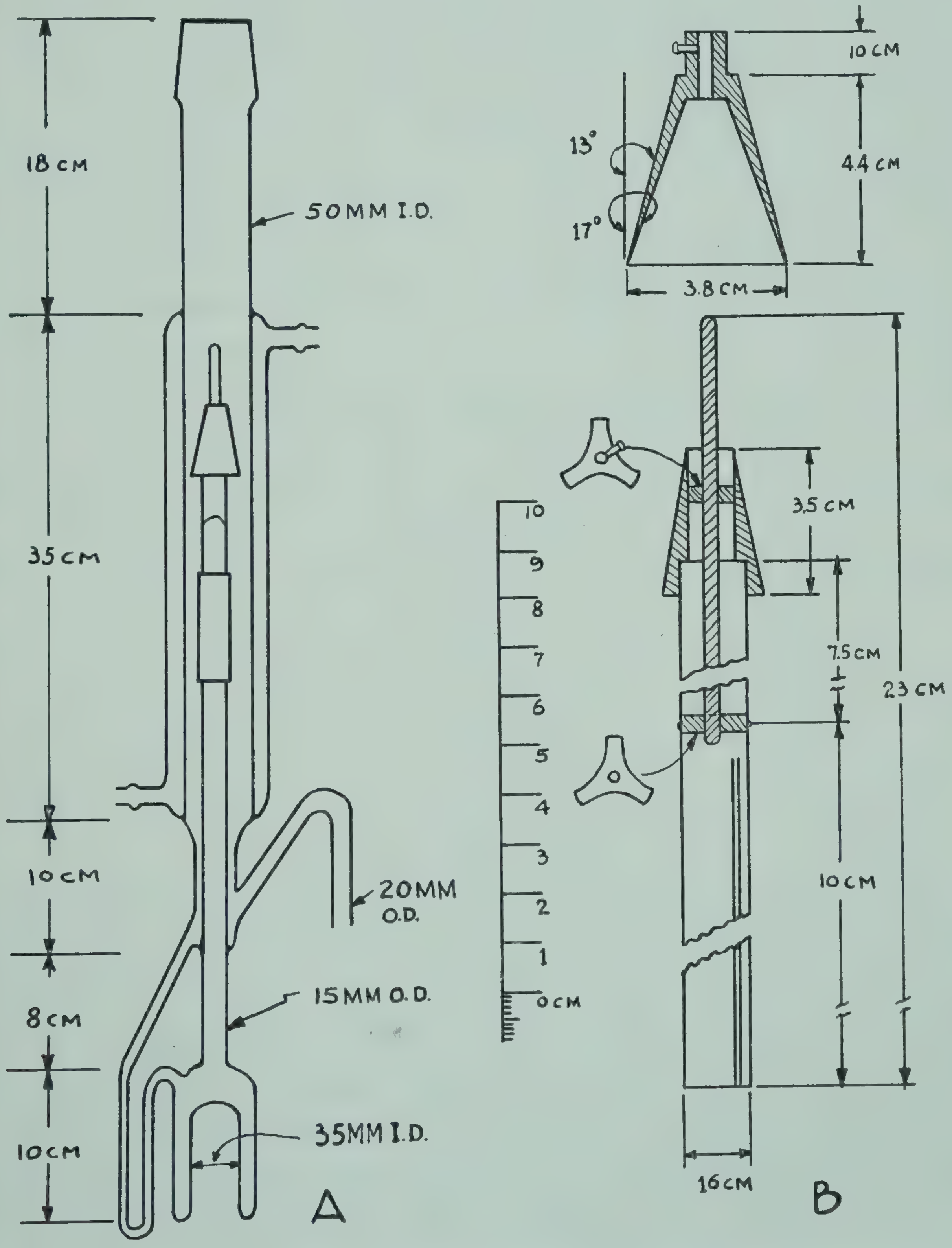
18 CM
50MM I.D.
35 CM
10 CM
20MM O.D.
8 CM
15MM O.D.
10 CM
35MM I.D.
A
10 CM
13°
4.4 CM
17°
3.8 CM
3.5 CM
10
9
8
7
6
5
4
3
2
1
0 CM
7.5 CM
23 CM
10 CM
16 CM
B

Figure 6. Combination metal-glass mercury diffusion pump.

Combination Metal and Glass Diffusion Pump. Estermann and Byck[7] designed and tested a mercury diffusion pump having a metal nozzle. The mercury boiler and the water condenser forming the body of the diffusion pump are of Pyrex glass, as is a section of the tube from the boiler. The metal nozzle and riser makes a friction fit over this glass tube from the boiler. Figure 6 is a drawing of this glass-metal mercury diffusion pump with details of the metal nozzle. In order to determine the dimension giving the maximum pumping speed, Estermann and Byck constructed the nozzle with a small clearance and tested it for pumping speed. The nozzle was then removed, turned down on the lathe, and again tested for pumping speed. The construction of the nozzle was such that by turning down the outer part of the nozzle on the lathe and at the same time cutting back the inner part to maintain relatively the same length, diffusion area was successively increased. In Table I are shown the results, which indicate that a diffusion area width of approximately 5.0 mm. produces the maximum pumping speed with this design.

TABLE I. Pumping Speeds of a Glass-Metal Diffusion Pump

Clearance, cm.	Area of diffusion region, cm.^{2}	Pumping speed, liters of air per second
0.1	1.5	3.0
0.2	2.9	5.0
0.33	4.7	8.5
0.55	7.4	13.0
0.63	8.4	9.5
0.71	9.2	8.4

Construction Data for a Two-Stage Diffusion Pump. A two-stage mercury diffusion pump of this same umbrella type is shown in Figure 7. Construction of the pump is basically the same as for those previously described, except for the nozzles. For these, one piece of 14 mm. O.D. tubing is prepared by blowing a 18 mm. O.D. elongated bulb and putting four 3 mm. I.D. holes above the bulb and equally spaced around the periphery of the tube, as shown in Figure 7A. A second section of tubing is prepared by

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[7] Estermann, I., and Byck, H., *Rev. Sci. Instr.* **3**, 482 (1932).

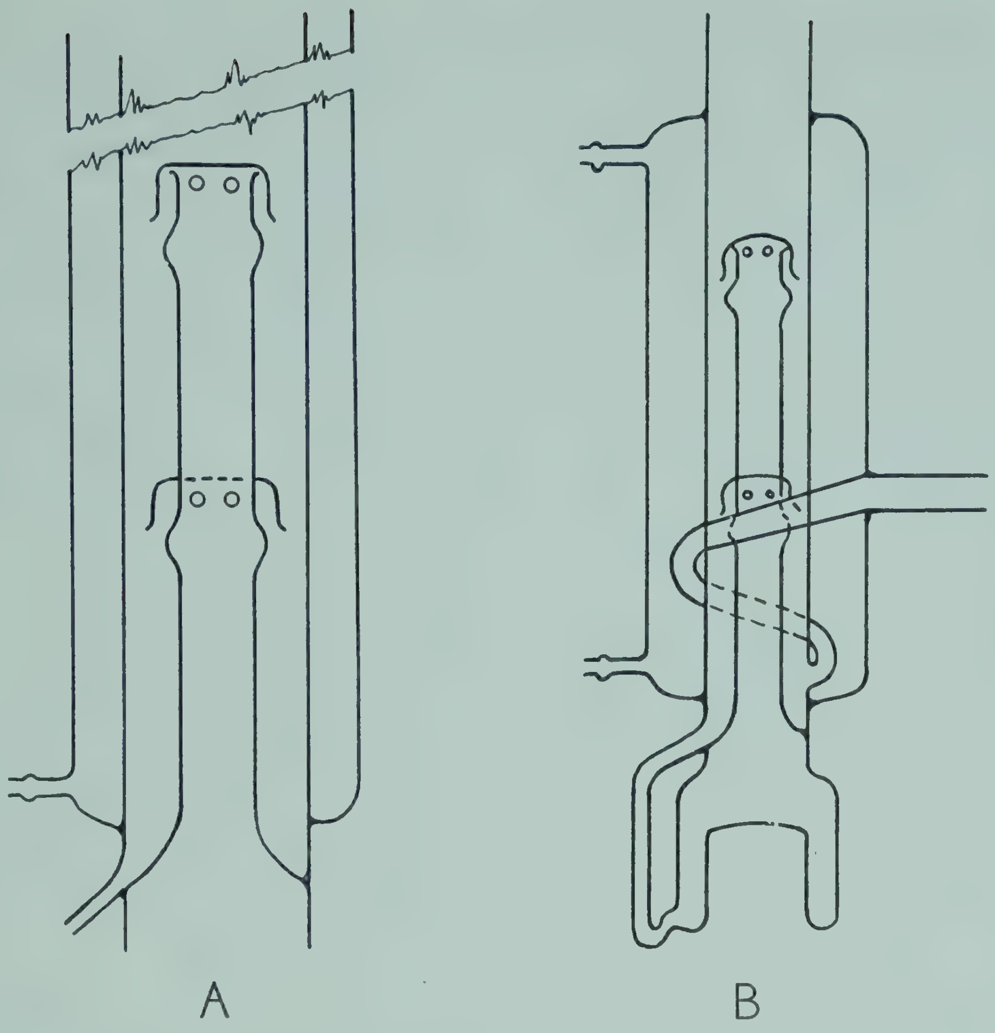

Figure 7. Two-stage mercury diffusion pump.

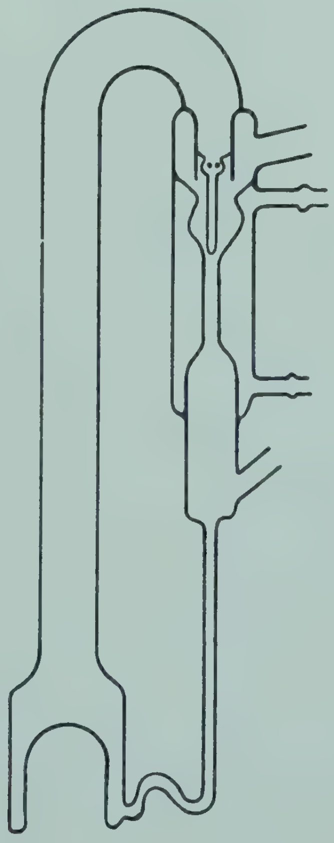

Figure 8. Kurth design mercury diffusion pump.

blowing a similar bulb and introducing three 3 mm. I.D. holes equally spaced around its periphery. The prepared tubes are then cut off about 15 mm. from the holes and the umbrellas held in place by a small wrapping of oven-baked asbestos paper tape. A ring seal is made through the first umbrella and the second prepared section is sealed into place, after which the ring seal is formed in the second umbrella but not blown out. The two-stage nozzle section should be supported with a wrapping of copper screening around each umbrella while it is being sealed in the condenser section and the pump finished according to the instructions for a single-stage mercury diffusion pump.

Mercury Down-Jet Diffusion Pump. A two-stage mercury diffusion pump of the Kurth design is shown in Figure 8. This pump has a pumping speed of 1.5 liters per second and is suitable for vacuum work where fast pumping speed is not necessary. As with the construction of umbrella-type pumps, all component parts are prepared before the pump is assembled. The condenser is made as previously described and the high-vacuum line sealed on just above the upper ring seal. The boiler is made and the nozzles are assembled as shown in Figure 8. The nozzle assembly is supported on center in the condenser by means of copper screen wrapping. The ring seal is then made and the tube which forms the mercury vapor line from the boiler is sealed in place and bent downwards. The boiler may then be sealed to this tube with a hand torch and the mercury return line connected. These last seals can be made more easily if the pump is conveniently clamped in a ring stand during the operation. In each step the apparatus should be carefully annealed as the work progresses and the pump finally annealed in an oven if possible. In Figure 9, complete description and dimensions are given for a larger and faster model of essentially this same pump.

Harrington[8] has taken a Kurth-type mercury diffusion pump and by the addition of a stopcock, a mercury trap, and a third jet, made it operate as a three-stage vapor pump and Sprengel pump combined. This pump, which has the speed of the former

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[8] Harrington, E. L., *Rev. Sci. Inst.*, **20**, 761 (1949).

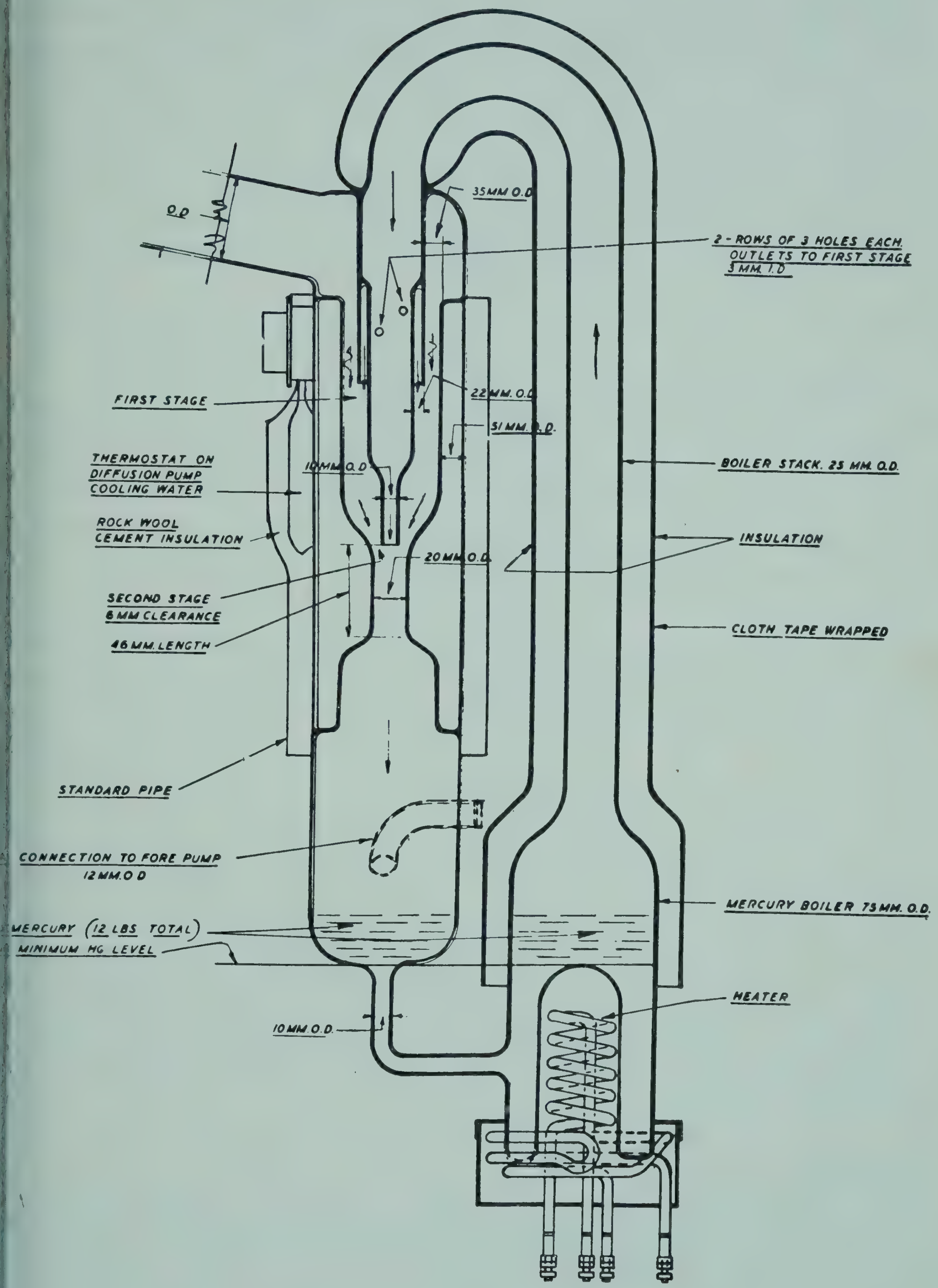
O.D
35MM O.D
2 - ROWS OF 3 HOLES EACH. OUTLETS TO FIRST STAGE 3 MM. I.D
FIRST STAGE
22MM. O.D
51MM. O.D.
THERMOSTAT ON DIFFUSION PUMP COOLING WATER
10MM. O.D
BOILER STACK. 25 MM. O.D.
ROCK WOOL CEMENT INSULATION
INSULATION
20MM. O.D
SECOND STAGE 8MM CLEARANCE
46MM. LENGTH
CLOTH TAPE WRAPPED
STANDARD PIPE
CONNECTION TO FORE PUMP 12MM. O D
MERCURY BOILER 75MM. O.D.
MERCURY (12 LBS TOTAL)
MINIMUM HG LEVEL
HEATER
10MM. O.D.

Figure 9. Large down-draft mercury diffusion pump.

and the trapping action of the latter, is shown in Figure 10. According to Harrington the only critical dimension is that of the third nozzle. This should be small to prevent more mercury from running over than can be condensed in the small bulb below. A nozzle of only 0.68 mm. I.D. was found satisfactory. The same Sprengel pump action can be applied to a standard commercial mercury diffusion pump of the Kurth design if it is properly modified.

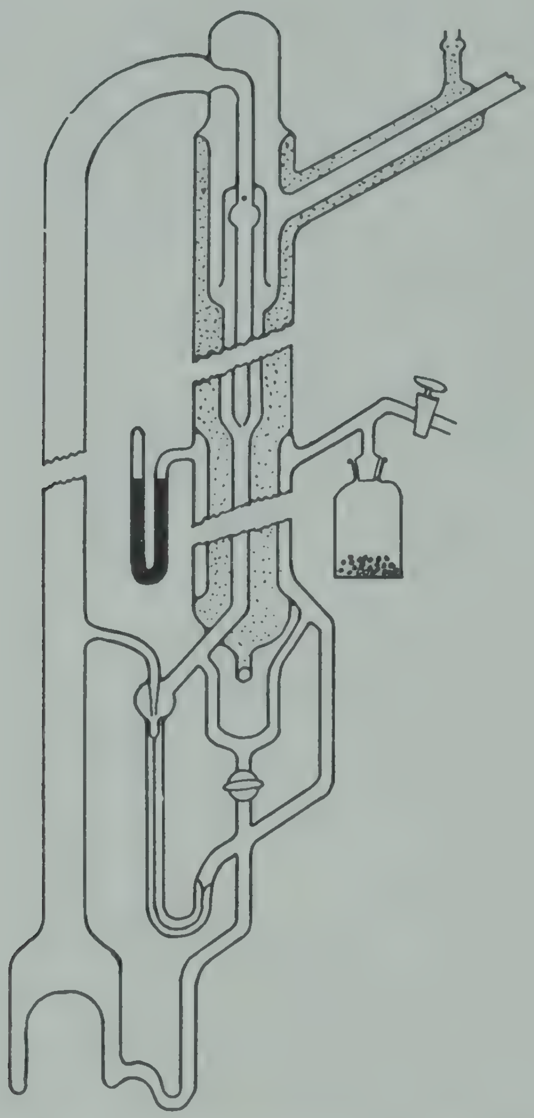

Figure 10. Modified Kurth (three-stage) mercury diffusion pump.

Heaters

Electrical heaters for mercury diffusion pumps may be obtained from the Eagle Electrical Co.[9] The wattage required to operate a two-stage umbrella-type pump will be from 350 to 400 watts and slightly more for the down-jet type pumps.

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[9] Eagle Electrical Co., Long Island City, New York.

Lagging

Mercury diffusion pumps with the overhead mercury vapor path should be lagged from the boiler to the top ring seal to prevent undue condensation of the mercury vapor. A satisfactory lagging can be made from Johns Manville asbestos cement #352. This is mixed with water and put on with a hand trowel. When it is dry or nearly dry a covering of asbestos cloth tape should be wrapped around it for further protection.

Oil Diffusion Pumps

The advantage of oil diffusion pumps over mercury diffusion pumps has generally been considered to be the elimination of the liquid air traps. Bearden[10] found that gases produced by decomposition of the oil gave rise to some deterioration of the vacuum and that to avoid this completely it was necessary to use a dry ice and alcohol trap. Martin and Hill[2] state that oil pumps without traps do not produce quite as low a pressure as properly trapped mercury pumps. According to Strong,[11] however, a charcoal trap is sufficient for ordinary use. Becker and Jaycox[12] claim to have obtained pressures as low as 10^{-8} mm. with oil diffusion pumps and a charcoal trap.

General Notes on Oil Diffusion Pumps. 1. The diameter of the delivery tube between the boiler and the jets should be as large as possible and the length kept as short as possible to avoid excessive condensation of the vapor and to maintain the reflux at a minimum. Lagging on the delivery tube will also help reduce refluxing. If the tube is too small in diameter or too long, the reflux may at times completely choke the delivery tube and result in momentary stoppage of the flow of vapor. This condition will result in a subsequently poorer vacuum.

2. The length of the condensing section is important. In operating glass pumps, a line of demarcation can be observed when the fore vacuum is of the order of 5×10^{-3} to 5×10^{-4}.

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[10] Bearden, J. A., *Rev. Sci. Inst.*, **6**, 276 (1935).

[11] Strong, *Procedures in Experimental Physics*, Prentice Hall, New York, 1939.

[12] Becker, J. S., and Jaycox, E. K., *Rev. Sci. Inst.*, **2**, 773 (1931).

This line of demarcation is known as the "Ring" and it forms at the point where the oil vapor strikes the condenser wall. The ring indicates that the pump is working, and it should hold steady at a distance at least equal to the diameter of the condenser at the front of the nozzle opening. It is desirable to use a fore pump which will maintain the ring at a distance of at least two diameters from the nozzle. The better the fore vacuum the further down the condenser the ring forms, up to five diameters. The length of the condenser should therefore be at least five times as long as the diameter. The position of the ring may be controlled to some extent by the heat applied to the boiler. If short condensers are used, the pump will be sensitive to temperature. Both the speed and the ultimate pressure may drop off after the critical temperature is only slightly in excess of the minimum necessary for the pump to operate. Properly designed oil diffusion pumps should not be temperature-sensitive.

3. Clearance between the nozzle and the condenser wall should exceed ⅛ in. to prevent oil droplets from closing off the space between the two.

4. The presence of water in the vacuum system should be carefully avoided. Oil absorbs considerable quantities of the gas or vapor to which it is exposed. Water if present in any appreciable quantity will completely stop the action of an oil diffusion pump and under normal operating conditions very little water vapor gets free and reaches the fore pump line. Should the pump oil become contaminated with water, the water vapor can be speedily removed by operating the pump at a low boiler temperature which scarcely vaporizes the oil. Under these conditions the water is vaporized and quickly removed by the fore pump, not, however, without an injurious effect on the oil.

5. Oxygen in the system at pressures greater than 1×10^{-2} mm. will cause the oil to burn. For this reason oil diffusion pumps should be always allowed to cool before opening the system to the atmosphere. Some oils will burn onto the heater coil if the system is accidentally opened to the atmosphere while the pump is operating. Should this happen the charred oil must be removed from the heater before the pump will operate properly.

Removal is accomplished by setting the heater Variac slightly lower than normal and allowing the pump to operate until the charred oil is burned from the heater. This may take only a few hours if charring was not excessive.

6. According to Boyle[13] silicone oils are superior to other liquids, both hydrocarbons and esters, in resistance to cracking and oxidation at high temperatures. Ionization gages should be protected from silicone vapors since, according to Boyle, when silicones decompose on metal surfaces they yield a layer of insulating material which can be removed only by heating to above 400° to 500° C. Apparently this insulating deposit will increase in time and ultimately render the gage useless as a pressure indicator.

7. It is desirable to use an oil vapor pump with two or more stages in series to obtain the maximum efficiency and speed, but these multiple stages should never be operated from the same boiler.

8. Fractionating oil diffusion pumps as designed by Hickman[14] eliminate to an extent the loss of vacuum due to decomposition of the oil. This fractionation is even more efficiently carried out in multiple-jet diffusion pumps so designed that the lighter components are gradually passed toward the backing

TABLE IIA. The Vapor Pressures of a Typical Range of Pumping Fluids*

Material	Pressure, mm, Hg
Apiezon A	10^{-5}
Apiezon B	10^{-7}
Butyle sebacate	2×10^{-5}
Amoil S	2×10^{-6}
Octoil	2×10^{-7}
Octoil S	5×10^{-8}
Silicone	5×10^{-8}
Mercury	3×10^{-9} at −78° C.
Mercury	2×10^{-27} at −180° C.

*Heavens, O. S., High Vacuum Practice, taken from Laboratory Practice 1, No. 3, p. 104, June (1952).

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[13] Boyle, R. A., John Dushman, ed., in *Vacuum Technique*. Wiley, New York, 1949.

[14] Hickman, K., *J. Appl. Phys.*, **11**, 303 (1940).

pump and the high-vacuum jet is supplied with the low-vapor-pressure fractions. A pump of this description is made by Distillation Products Co.

Table IIA gives the values of vapor pressure for a typical range of boiling fluids; the improvement in the ultimate pressure attained with multiple-stage pumps over single-stage pumps is shown in Table IIB.

TABLE IIB. Ultimate Pressures for Standard Commercial Boiling Fluids

Fluid	Pressure (optimum) mm. Hg	Pump	Backing pressure, mm. Hg
Butyl sebacate	2.5×10^{-5}	Single-boiler	0.1
	10^{-5}	Multi-boiler	0.15
Amyl sebacate	10^{-5}	Single-boiler	0.1
(Amoil-S)	2×10^{-6}	Multi-boiler	0.17
Ethyl hexyl phthalate	5×10^{-6}	Single-boiler	0.1
(Octoil)	2.5×10^{-7}	Multi-boiler	0.25
Ethyl hexyl sebacate	10^{-6}	Single-boiler	0.1
(Octoil-S)	5×10^{-8}	Multi-boiler	0.2

Construction Data. 1. A single-stage oil diffusion pump is shown in Figure 11. In this pump the heater is immersed directly in the oil, resulting in very accurate control of the temperature of the oil. This pump depends on air cooling, although it can be made with a water jacket or a water-cooled coil of copper tubing

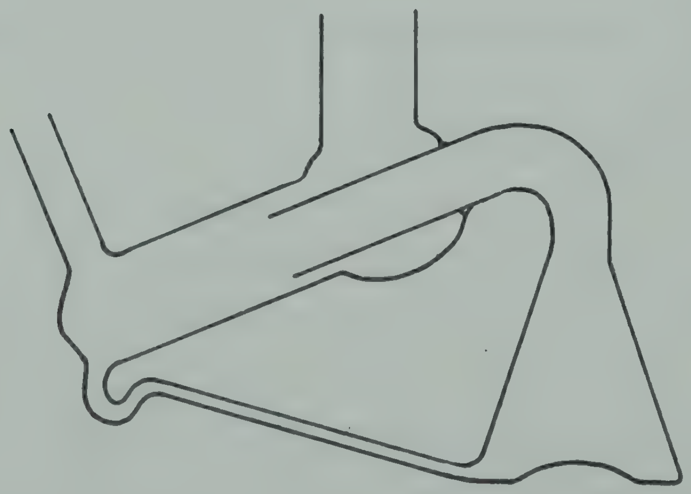

Figure 11. Single-stage oil diffusion pump.

or thin rubber tubing may be wrapped around the condensing section.

2. A single-stage four-nozzle oil diffusion pump of high pumping speed is shown in Figure 12. This pump was designed by Zobel[15] and described in the *Review of Scientific Instruments.* It has a rated pumping speed of 41 liters per second at 5×10^{-4} mm. This pump is also cooled by wrapping coils of thin rubber tubing for water cooling or it may have a water jacket blown in place. It is recommended that the boiler and throat be covered with a heavy coating of asbestos to reduce the necessary heat input to the oil.

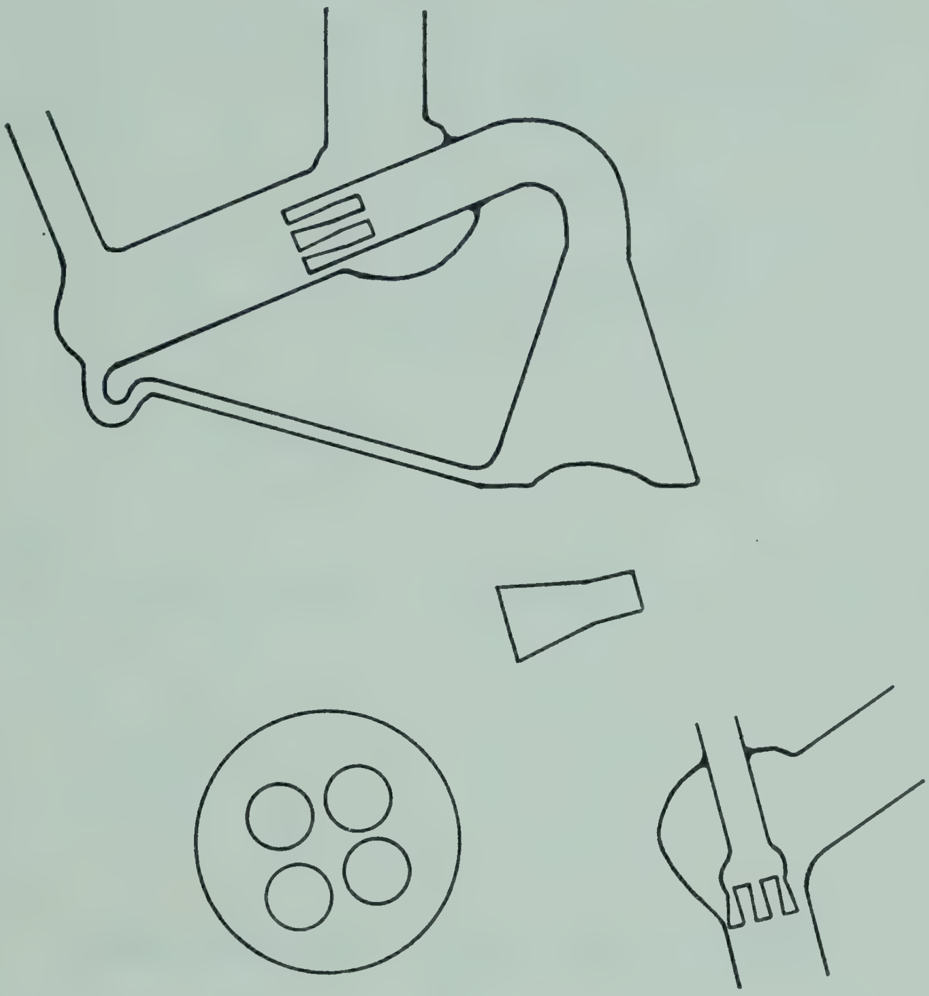

Figure 12. Four-jet oil diffusion pump.

3. A single-stage seven-nozzle pump of similar design is shown in Figure 13. This pump may also be cooled by coils of thin rubber or it may have a water condenser blown in place. The

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[15] Zobel, *Rev. Sci. Instr.*, **7**, 54 (1935).

pumping speed of this seven-jet pump is rated at 76 liters per second at 5×10^{-4} mm. with a heat input of around 300 watts.

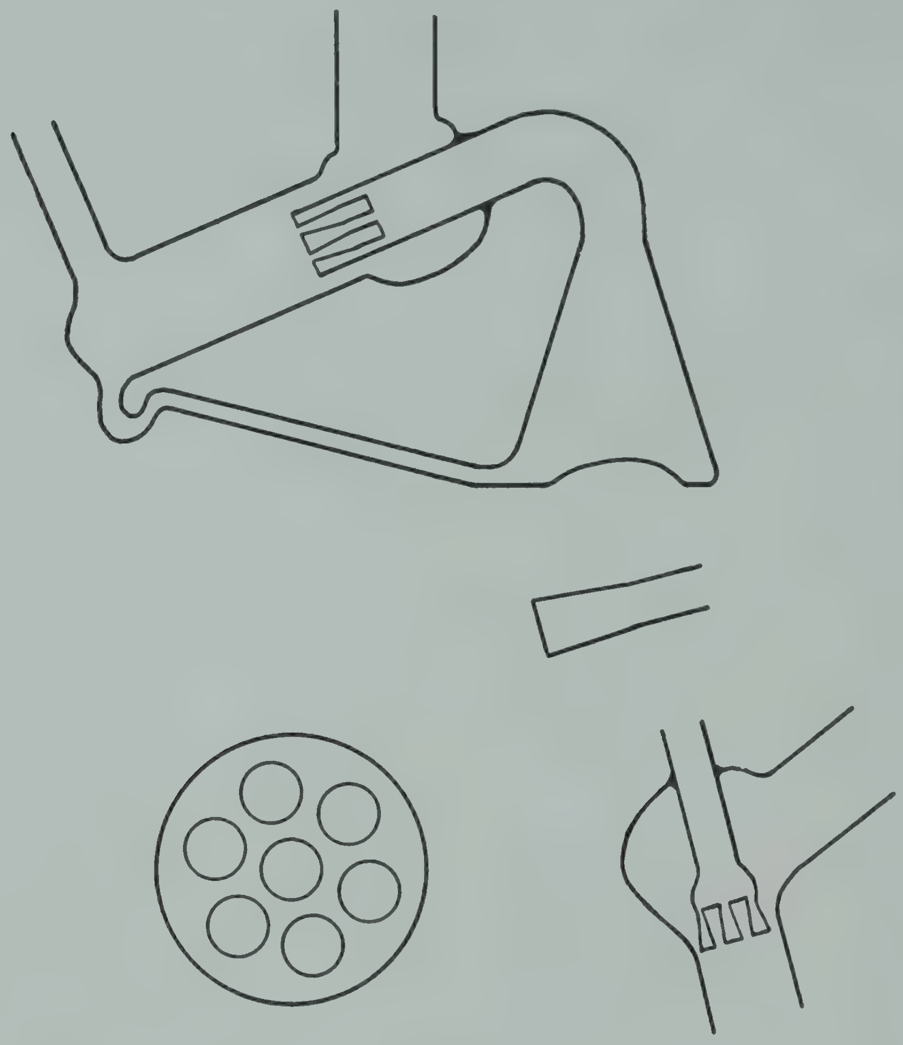

Figure 13. Seven-jet oil diffusion pump.

4. Two oil-diffusion pumps shown in Figure 14, when connected in series, produce a vacuum of better than 10^{-7} mm. at a pumping speed of about 1.5 liters per second. The heat input of these pumps is about 150 watts each.

5. Diffusion pump boilers. Some of the possible diffusion pump boiler designs are shown in Figure 15. A round boiling flask heated by means of a Glas-Col heating mantle is pictured in Figure 15A. Figure 15B shows a boiler designed for use with a standard Glocoil- electric heater unit. The boiler shown in Figure 15C is an immersion-type electric coil heater suitable for oil diffusion pumps. This type of heater gives very efficient and accurate control of boiling.

It is not necessary to lag the boiler of mercury diffusion pumps of the umbrella design; however, the boilers of all oil dif-

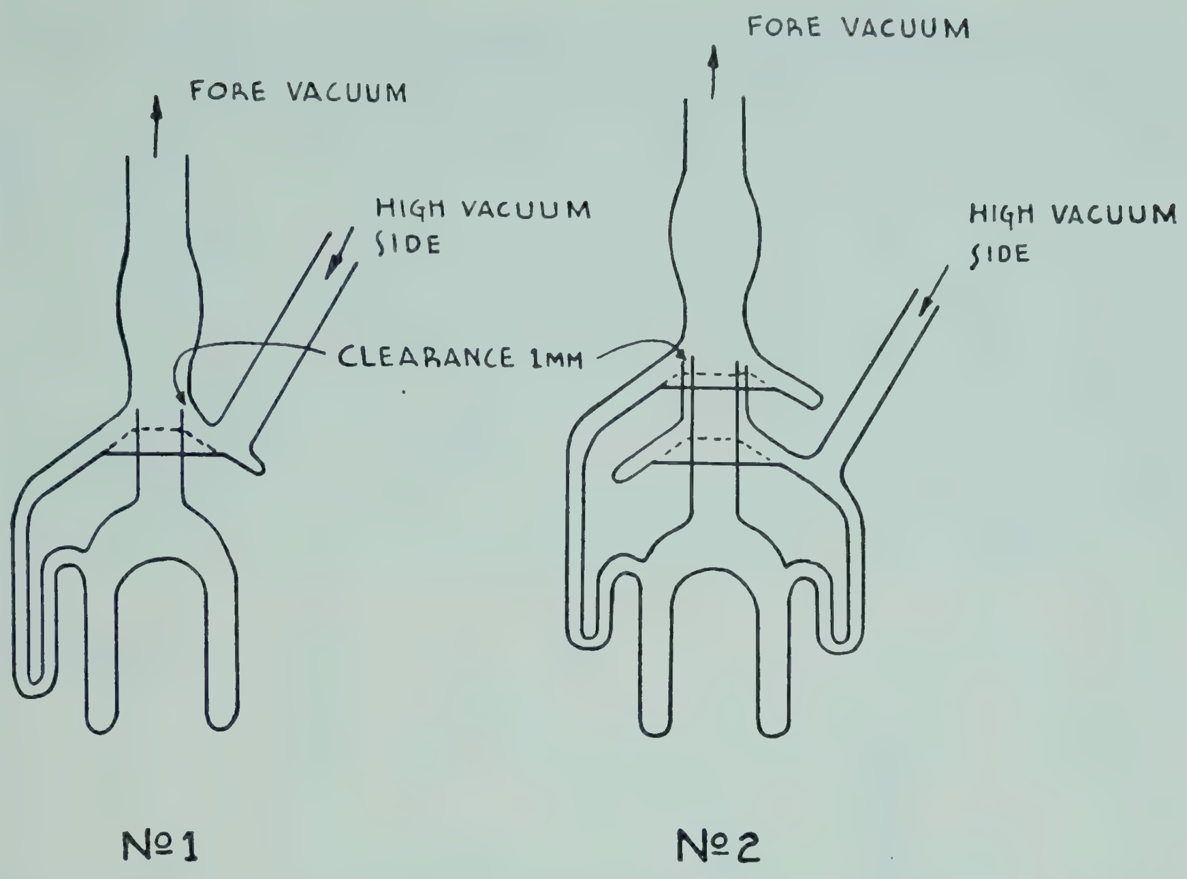
FORE VACUUM
HIGH VACUUM SIDE
CLEARANCE 1MM
FORE VACUUM
HIGH VACUUM SIDE
№1
№2

Figure 14. Two oil diffusion pumps for in series mounting.

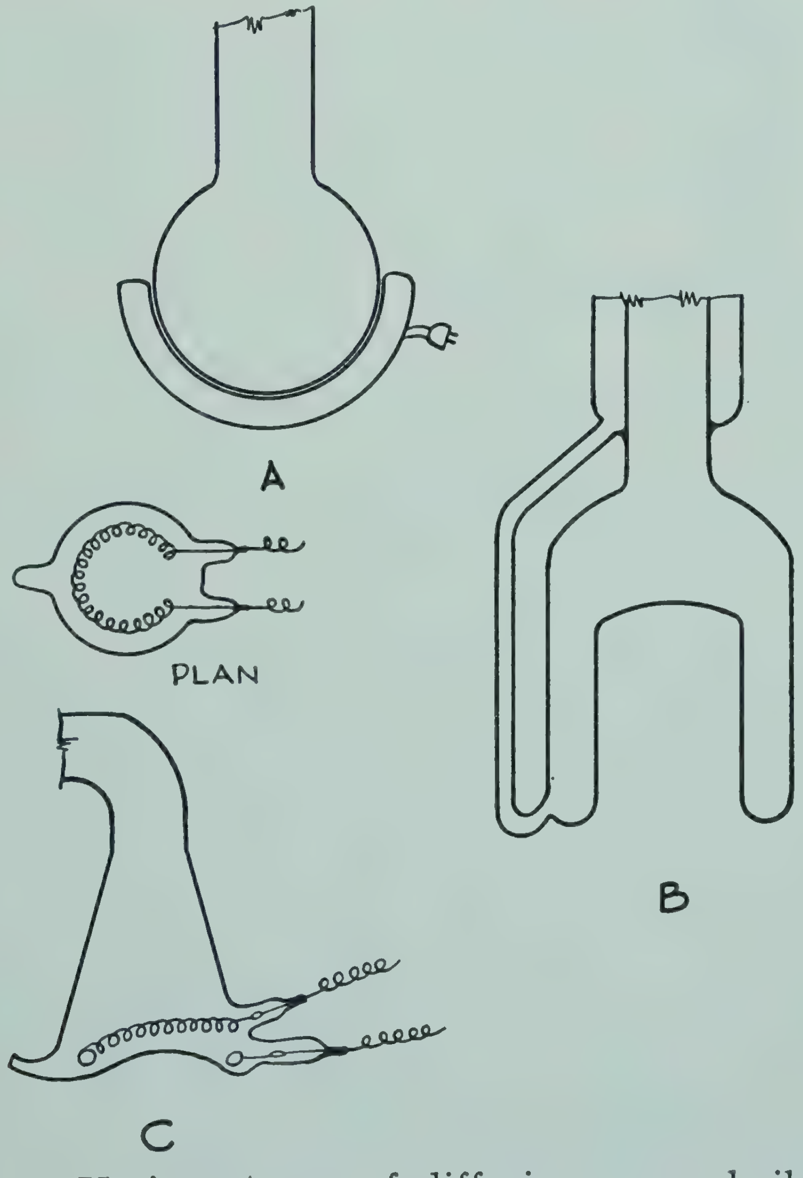

Figure 15. Various types of diffusion pump boilers.

fusion pumps should be lagged with at least a ½-in. coating of asbestos.

Determining Pumping Speeds. The apparatus shown in Figure 16 may be used for measuring the pumping speed of diffusion pumps by means of a moving column of mercury. This should be assembled directly over the diffusion pump or as near the pump as possible. The pressure in the pump is measured by a McLeod gage and may be adjusted to any desired value by regulating the air which enters the pump through stopcock 1. With stopcock 2 closed and stopcocks 1 and 3 open, the mercury levels A in the capillary tube and leveling tube are brought even. The pressure in the pump is regulated to any desired value by slightly opening the slow leak (stopcock 2) which regulates the admission of air into the pump by way of the capillary tube. When several readings of the McLeod gage indicate that the system has equilibrated, the tests can be started using the following procedure. Stopcock 3 is closed and the time necessary for the mercury column to rise from A to B in the capillary is taken with a stopwatch. When the level reaches B, stopcock 1 is closed and the

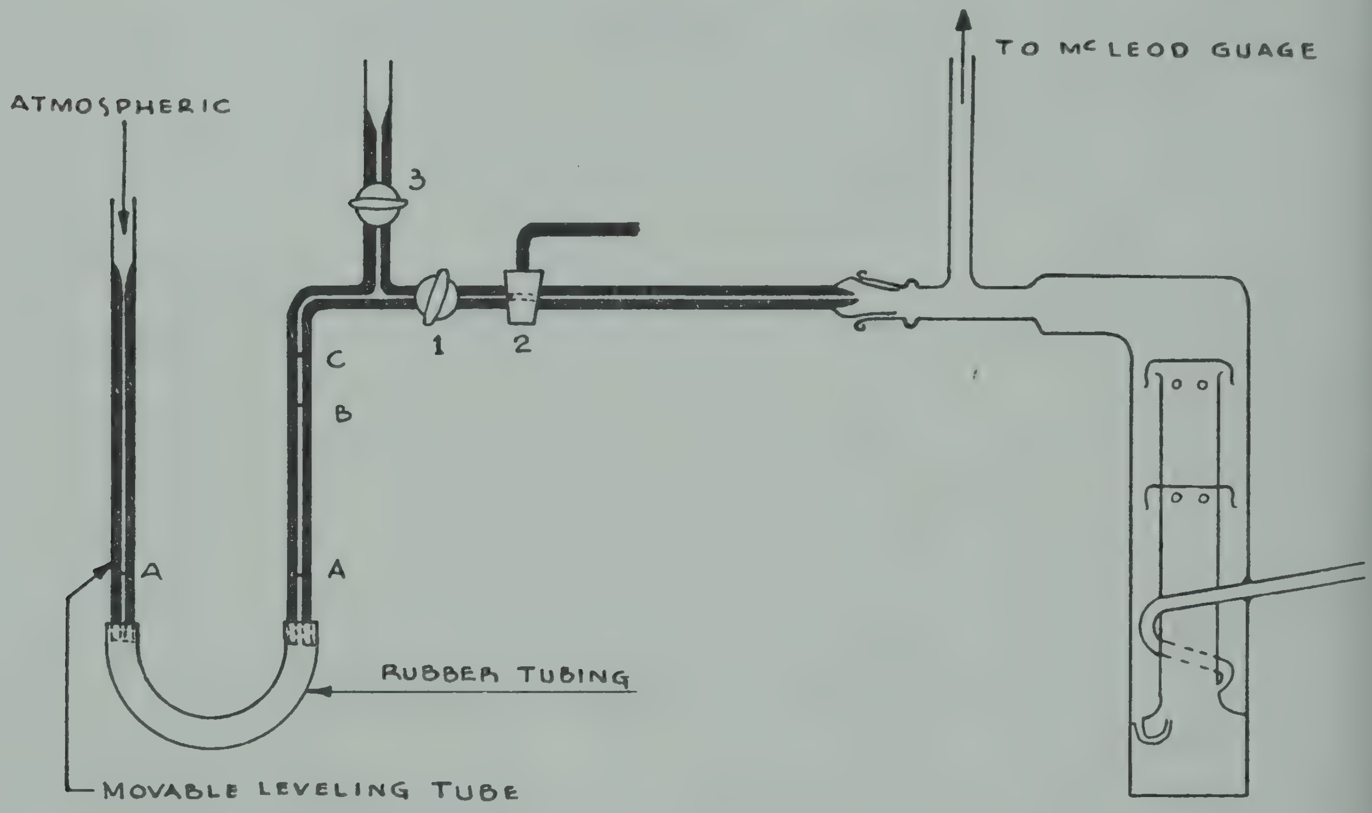

Figure 16. Apparatus for determining the pumping speed of diffusion pumps.

mercury columns in the capillary and leveling tube are brought even again, resulting in a new level, C. When measured from A, this gives the amount of gas pumped out. Then if:

V = Volume
A = Area of cross section of capillary
T = Time in seconds
H = Height of capillary
L = Liters per second
P = Pressure at which measurement was made

we have

$$V = \frac{A \times H}{T \times 1000} = L \times \frac{760}{P}.$$

Vapor Traps

Liquid-Nitrogen Traps. Vacuum systems which employ mercury diffusion pumps usually use a liquid-nitrogen trap. The temperature of commercial liquid nitrogen generally is about —180° C., at which temperature the vapor pressure of mercury is about 1.7×10^{-27} mm (see Table III).

Dry Ice and Alcohol Traps. A trap cooled by dry ice and alcohol, which produces a temperature of $-78°$ C., is sufficient in some cases. The vapor pressure of mercury at $-78°$ C. is 3.2×10^{-9} mm. It should be noted that dry ice and alcohol will not remove water vapor, since the vapor pressure of ice is about 1 micron at $-78°$ C. In practice it is advisable not to apply a refrigerant to the trap until the pressure in the system is below 10 microns, thus avoiding trapping water and other volatile gases. Traps which are cooled prematurely will in effect create a "virtual" leak. Water in particular will condense above the level of the refrigerant and slowly evaporate as pumping continues, giving the effect of a slow leak.

Notes on Refrigerants. Methyl alcohol, acetone and ether are commonly used refrigerants in dry ice traps. All three have one fault in common, that is, they foam when the dry ice is added too quickly. Isopropyl alcohol, on the other hand, will foam very

TABLE III. Pressures of Saturated Mercury Vapor*

Temperature	—180° C.	—78° C.	—40° C.	—20° C.
Vapor pressure	1.7×10^{-27}	3.2×10^{-1}	1.7×10^{-6}	2.2×10^{-5}
Temperature	—10° C.	0° C.	10° C.	
Vapor pressure	6.5×10^{-5}	1.9×10^{-9}	4.9×10^{-4}	
Temperature	20° C.	30° C.		
Vapor pressure	1.2×10^{-3}	2.8×10^{-3}		

*Denotes pressures above solid mercury.

little even when it is at room temperature, probably owing to the alcohol's becoming viscous as it cools. The melting point of isopropyl alcohol is —88.5° C., compared to —95° C. for acetone and —97.8° C. for methanol.

For traps which have to have the refrigerant removed and replaced at intervals, a mixture of 50 % chloroform and 50 % carbon tetrachloride will be advantageous. This will cause the dry ice to float in the liquid instead of dropping and packing at the bottom of the Dewar.

Potassium Traps. According to Poindexter,[16] potassium traps are as efficient as liquid air in arresting mercury vapor. This fact is especially useful when supplies of liquid air are not readily available or when the vacuum system must be run continuously over a long period of time. It has been stated that potassium will absorb more than its own weight of mercury—a process which takes several weeks under ordinary circumstances. Moderate exposure of potassium to air does not affect its capacity for absorbing mercury vapor. The technique of preparing a potassium trap is as follows:

About 4 oz. of clean potassium is heated in a Pyrex tube as shown in Figure 17A. The Bunsen flame used for heating the potassium will drive off hydrocarbons from the solution in which the metal is customarily kept. When the metal itself begins to distill, it is poured into tube B, where it solidifies. Most of the oxide will be found to have been left in tube A. Tube B is then sealed to a vacuum system as shown in Figure 17C. The potassium is then heated until the large evolution of gas nearly ceases. The metal in B is then heated until all the metal is driven into C, at which time B is sealed off. The metal in C is next heated until it

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[16] Poindexter, F. E., *Phys. Rev.*, **28**, 208 (1926).

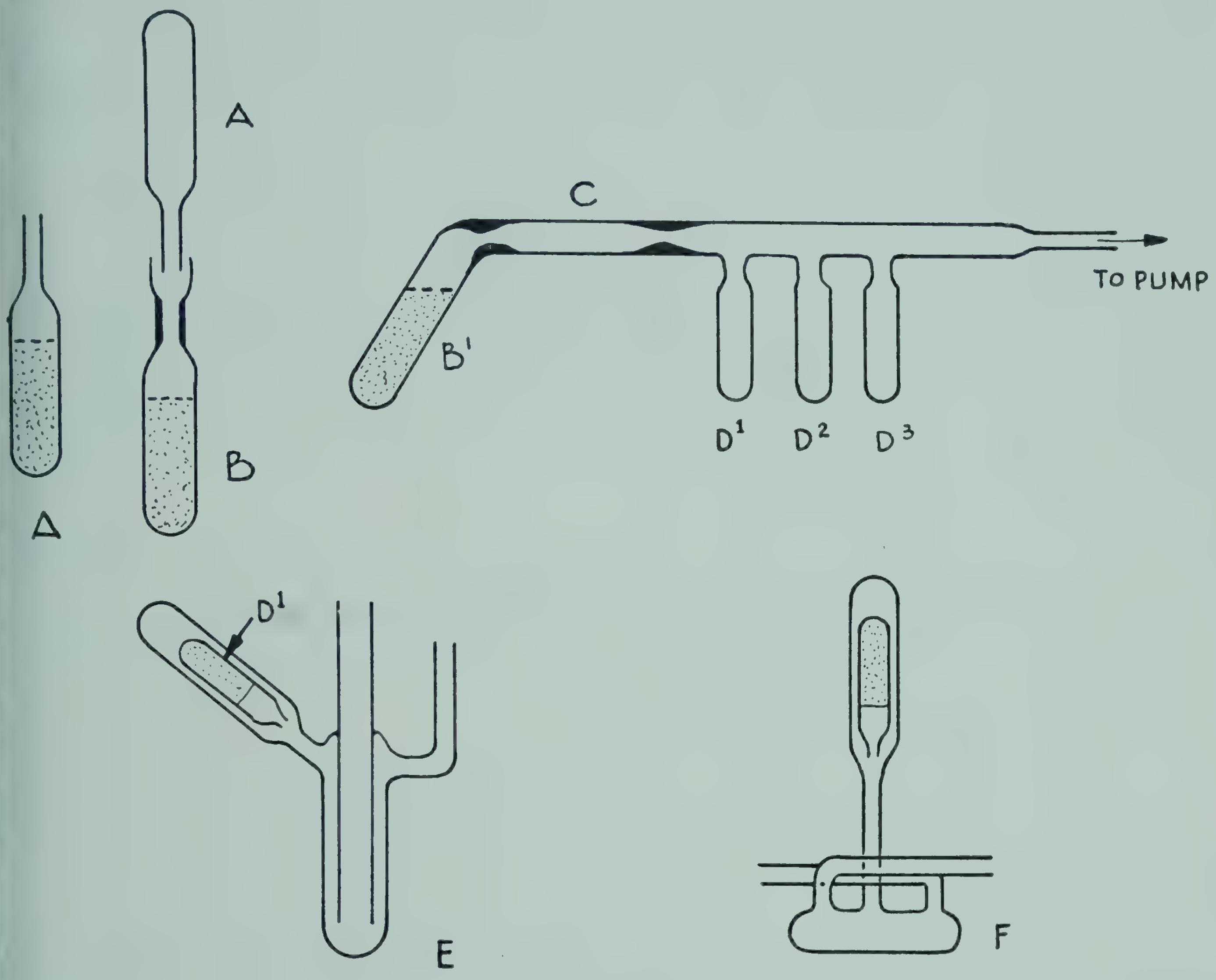

Figure 17. Potassium traps.

liquefies and then, by tilting the apparatus, the small containers, D1, D2, D3, are filled and sealed off. When the mercury vapor trap is to be lined with potassium, one of these containers is opened and sealed in a tube as shown in Figure 17E. The trap is sealed into a vacuum system and evacuated. As soon as a good vacuum has been reached, the potassium is heated and driven into the trap, where by further heating it is vaporized and forms an absorbent coating over the walls of the flask. Hunten, Woonton, and Longhurst[17] reported on a special trap which is self-reactivating even after admission of moist air. In this trap alloys of sodium and potassium containing only 3 % to 5 % of potassium were used. Hunten reported that these alloys, which are liquid at room temperature, were more satisfactory than either sodium or potassium alone. Purification of the alloy and its distillation into the capsule followed the method described by Poindexter. However, the trap shown in Figure 17F was adopted and found to be superior to all others tried. When the trap is to be filled the bottom of the capsule is broken and the capsule lowered into a larger diameter glass tubing which is sealed to the trap. The metal is then allowed to flow through the constriction until the trap is half full, after which the trap is sealed off at the constriction. An alternate method of filling the trap is to use oil-free sodium, which needs no purification and can, therefore, be inserted directly into the trap.

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[17] Hunten, K. W., Woonton, G. A., and Longhurst, E. C., *Rev. Sci. Inst.*, **18**, 842 (1947).

It was reported that a vacuum system protected by this alkali metal alloy trap was repeatedly pumped from atmospheric to 5×10^{-7} mm. over a period of 22 days and during this period the traps required no attention.

Charcoal traps. Becker and Jaycox[18] reported that the addition of a simple trap containing commercial activated charcoal would permit pressures of 10^{-8} mm. to be reached with oil diffusion pumps. Figure 18 shows the design for several types of traps. Figure 18A is a simple dry ice trap filled with activated charcoal. Both the inlet and outlet tubes are closed off with glass wool to prevent the charcoal from being carried to other parts of the system.

Figure 18B shows two nickel screen baskets. The section on the left is a solid core of charcoal. The outside diameter of the nickel screen basket containing the charcoal is 1.4 times the radius of the inside of the glass tubing into which it slides. The basket is centered and held in position in the glass tube by means of nickel wire projections soldered in place. (See Fig. 18B.) The section on the right is hollow and allows the gas to pass through it. With the two sections of the trap in position as shown no gas molecules can pass through or around them without coming into contact with the charcoal.

[18] Becker, J. A., and Jaycox, E. K., *Rev. Sci. Inst.*, **2** 773 (1931).

A single basket of charcoal may be used as shown in Figure 18C. This is the most efficient. It has the highest resistance and therefore the lowest pumping rate is obtainable through this type.

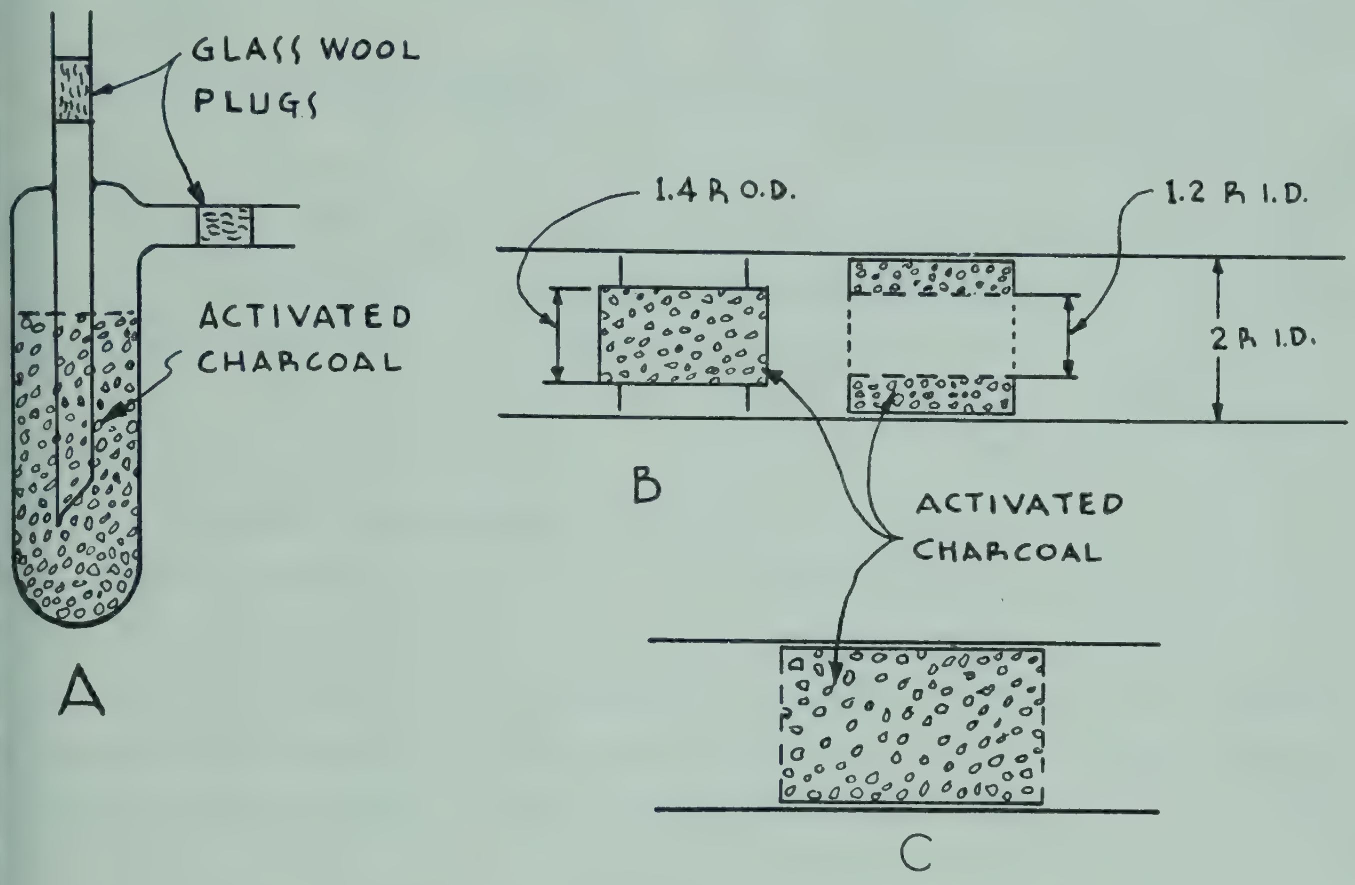

Figure 18. Charcoal traps.

To use a charcoal trap the vacuum system and trap should first be evacuated by means of the fore pump. When the pressure has been reduced somewhat, the charcoal trap should be baked at 400° C. until the excessive evolution of gas subsides. When two charcoal traps are used, one should be located between the system and the diffusion pumps and the second between the diffusion pumps and the fore pump. The trap on the low-vacuum side will need only a few minutes baking out at 400° C., and this need not be done each time the vacuum system is used. Table IV shows the amounts of gas sorbed by activated charcoal at the temperatures of ice and liquid nitrogen. The volumes of gas are in c.c. reduced to 760 mm. Hg pressure and 0° C.

The remarkable sorption by charcoal is due to its large surface area. According to Martin,[2] the surface may be as great as 2500 m^2. per gram.

TABLE IV. Sorption by Activated Charcoal*

Gas	O_2	CO_2	A	N_2	H_2	He
Sorption by 1 cc. of charcoal at 0° C.	18	21	12	15	4	2
Sorption by 1 cc. of charcoal at —185° C.	230	190	175	155	135	15

*Encyclopaedia Brittanica **16**, 751 (1911).

Obtaining a vacuum by means of a charcoal trap: The residual gases in a vacuum system which does not include a diffusion pump may be absorbed in a charcoal trap by the following technique.

The fore pump is turned on and the vacuum system is evacuated while the charcoal trap is degassing. When the ultimate pressure of the pumps is reached, the system is sealed off from the fore pump and the charcoal trap is immersed in liquid nitrogen. Dushman[19] reported an experiment in which a pressure of less than 5×10^{-9} mm. was obtained by this method. Since a mixture of charcoal and liquid oxygen is explosive, any glass

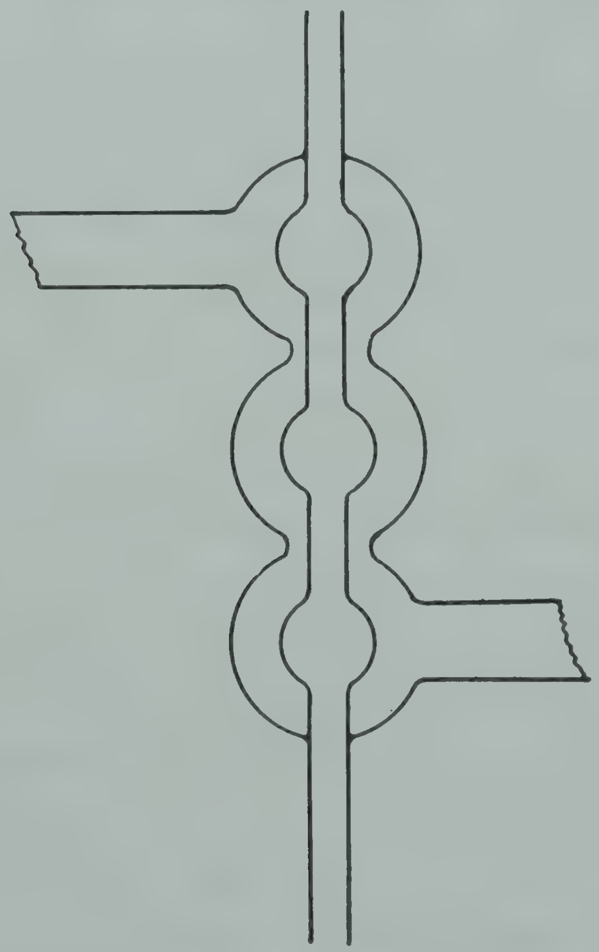

Figure 19. Water trap for oil diffusion pumps.

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[19] Dushman, S., *J. Franklin Inst.*, **211**, 721 (1931).

tube used to hold the charcoal should be protected by copper sheathing to minimize the danger of breakage.

Water Traps. Hickman[20] designed the water trap shown in Figure 19 for use with diffusion pumps using Octoil.[21]

MEASUREMENT OF PRESSURE

In considering the methods of measuring pressures, the various gages may be separated into six categories as follows:

1. Those which depend on electrical discharge through gases.
2. Those which depend on Boyle's law.
3. Those which depend on molecular bombardment due to temperature differences.
4. Those which depend on heat transfer.
5. Those which depend on the frictional drag of molecules on a moving surface.
6. Those which depend on the electrical conductivity of gases.

The choice of the type of gage to be used will depend on the conditions under which it will operate; i.e., is it necessary that the readings be continuous, or are intermittent readings satisfactory, and will the pressure variation be rapid or slow?

Discharge Tube (Pressure Range, 10 to 10^{-2} mm.Hg)

In some cases a simple discharge tube will give a sufficient indication of the pressure. Figure 20 shows a tube designed by Burrows.[22] The tube is operated by a high-voltage Tesla coil. According to Burrows the discharge will vary according to the pressure in the following manner:

0.5 cm. diameter column of glow discharge	10 mm. Hg
First visible striations	1.5 mm. Hg
Striations separated, 1 cm. apart	0.5 mm. Hg
Green fluorescence on inside walls	0.01 mm. Hg
Blackout (under vacuum conditions)	0.001 mm. Hg or less

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[20] Hickman, K. E. D., *J. Franklin Inst.*, 221, 338 (1936).

[21] This oil is obtained from J. Biddle & Co., Philadelphia, Pennsylvania.

[22] Burrows, G., *J. Sci. Inst.* **20**, 21 (1943).

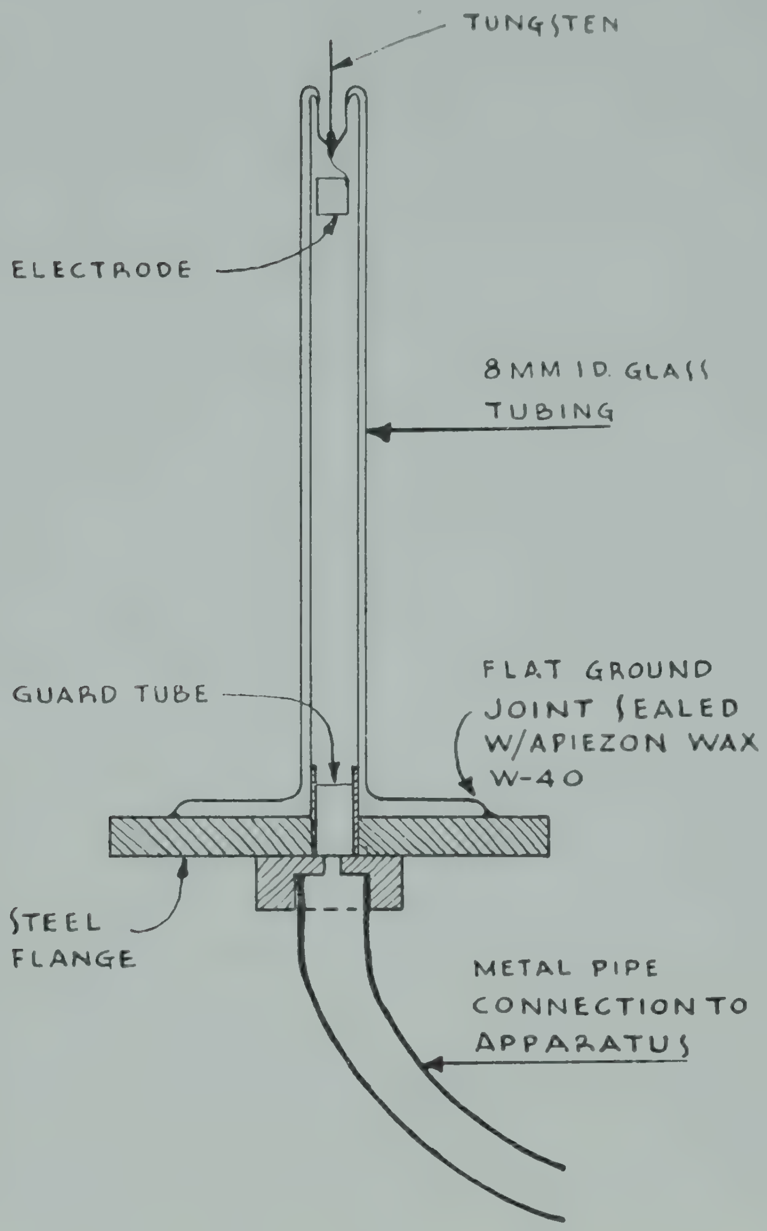
TUNGSTEN
ELECTRODE
8 MM I.D. GLASS TUBING
GUARD TUBE
FLAT GROUND JOINT SEALED W/APIEZON WAX W-40
STEEL FLANGE
METAL PIPE CONNECTION TO APPARATUS

Figure 20. Discharge tube.

It is also possible to tell the nature of the gas present with a discharge tube by the color of the discharge. See Table V.

TABLE V. Appearance of Discharges in Gases and Vapors at Low Pressures

Gas	Negative glow	Positive glow
Air	Blue	(Reddish)
Nitrogen	Blue	Yellow (red gold)
Oxygen	Yellowish white	Lemon
Hydrogen	Bluish pink (bright blue)	Pink (rose)
Helium	Pale green	Violet red
Argon	Bluish	Deep red (violet)
Neon	Red-orange	Red-orange (blood red)
Krypton	Green	—
Xenon	Bluish white	—

TABLE V. (Continued)

Gas	Negative glow	Positive glow
Carbon monoxide	Greenish white	(White)
Carbon dioxide	Blue	(White)
Methane	Reddish violet	—
Ammonia	Yellow-green	—
Chlorine	Greenish	Light green
Bromine	Yellowish green	Reddish
Iodine	Orange-yellow	Peach blossom-colored
Lithium	Bright red	—
Sodium	Yellowish green	Yellow
Potassium	Green	Green
Mercury	Green (goldish white)	Greenish blue (greenish)

— Indicates no distinctive color given.

Lehrbuch der Physik, IV.
Martin, L. H., and Hill, R. D., *A Manual of Vacuum Practice*, p. 112. Melbourne University Press, Melbourne, 1946.

McLeod Gage

The McLeod[23] gage, which was designed in 1874, is used extensively in high-vacuum work and is a convenient means of calibrating other low-pressure gages. McLeod gages are thoroughly reliable for permanent gases in ranges of 10^{-1} to 10^{-5} mm. of mercury and may be read to 10^{-6} mm., although in this range they are much less reliable.

Dimensions. For a vacuum in the range of 10^{-4} to 10^{-5} mm. the McLeod gage is usually made with a flask 200 to 300 ml. in volume and the capillary diameter is 1 mm. Increasing the volume of the flask to 500 ml. will make it possible to read the pressure to 10^{-6} mm., but this entails the use of considerably more mercury and the weight becomes a factor. The maximum convenient volume to be considered is 500 ml. Capillary tubes may be used with as small an inside diameter as 0.5 mm.; if smaller capillary tubes than this are used, the mercury thread tends to separate when the mercury level is lowered. Dirty mercury also has a tendency to stick and split in small capillary tubes as the level of the mercury is lowered.

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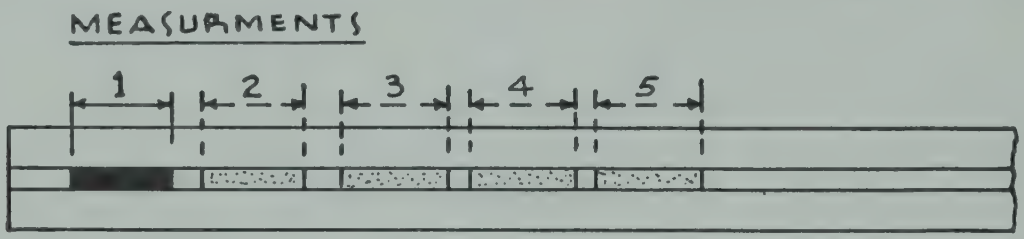
[23] McLeod, *Phil. Mag.*, **47**, 110 (1874); quoted in G. W. C. Kaye, *High Vacuum*, Longmans, Green, London, 1927.

Selecting Capillary Tubing. Several 3-foot sections of capillary tubing are selected and carefully cleaned and dried. A small amount of clean mercury is then drawn up into the capillary and measured. Measurement should be made with a ruler which can be read accurately to 0.1 mm., and a jeweler's magnifying eye loop should be used to increase the accuracy with which the measurements can be made. After each segment is measured and the results recorded, the thread of mercury is moved down the capillary and a new measurement taken. (See Figure 21.) This is



MEASURMENT	LENGTH	
1	5.3 CM	
2	5.25 CM	
3	5.20 CM	
4	5.15 CM	SEALED TO BULB
5	5.15 CM	
6	5.20 CM	
7	5.15 CM	SIDE ARM
8	5.15 CM	
9	5.15 CM	
10	5.20 CM	
11	5.20 CM	

Figure 21. Selecting the capillary for making a McLeod gage.

repeated for the full length of the capillary, or until variation in the bore of the capillary indicates it will be unsatisfactory. It is not usually too difficult to find a capillary with a variation of not over 1 %. The mercury is then removed from the capillary and weighed. From the weight of the mercury and its temperature at the time the measurements were made, it is simple to calculate the inside diameter of the capillary and the volume per millimeter of length. Precision-bore capillary tubing may be used without step No. 1.

From the selected length of capillary tubing two sections are chosen with as nearly identical bores as possible, so as to avoid inaccurate readings due to an unequal capillary depression of mercury. The capillary section which is to be sealed to the flask is first sealed to a piece of 8 to 9 mm. standard-wall tubing. This small section of standard-wall tubing reduces the danger of the capillary tubing's being broken from the flask by a surge of mercury in the event the mercury is allowed to rush into the flask too rapidly. Care should be exercised in making the seal between the capillary tubing and the standard-wall tubing to see

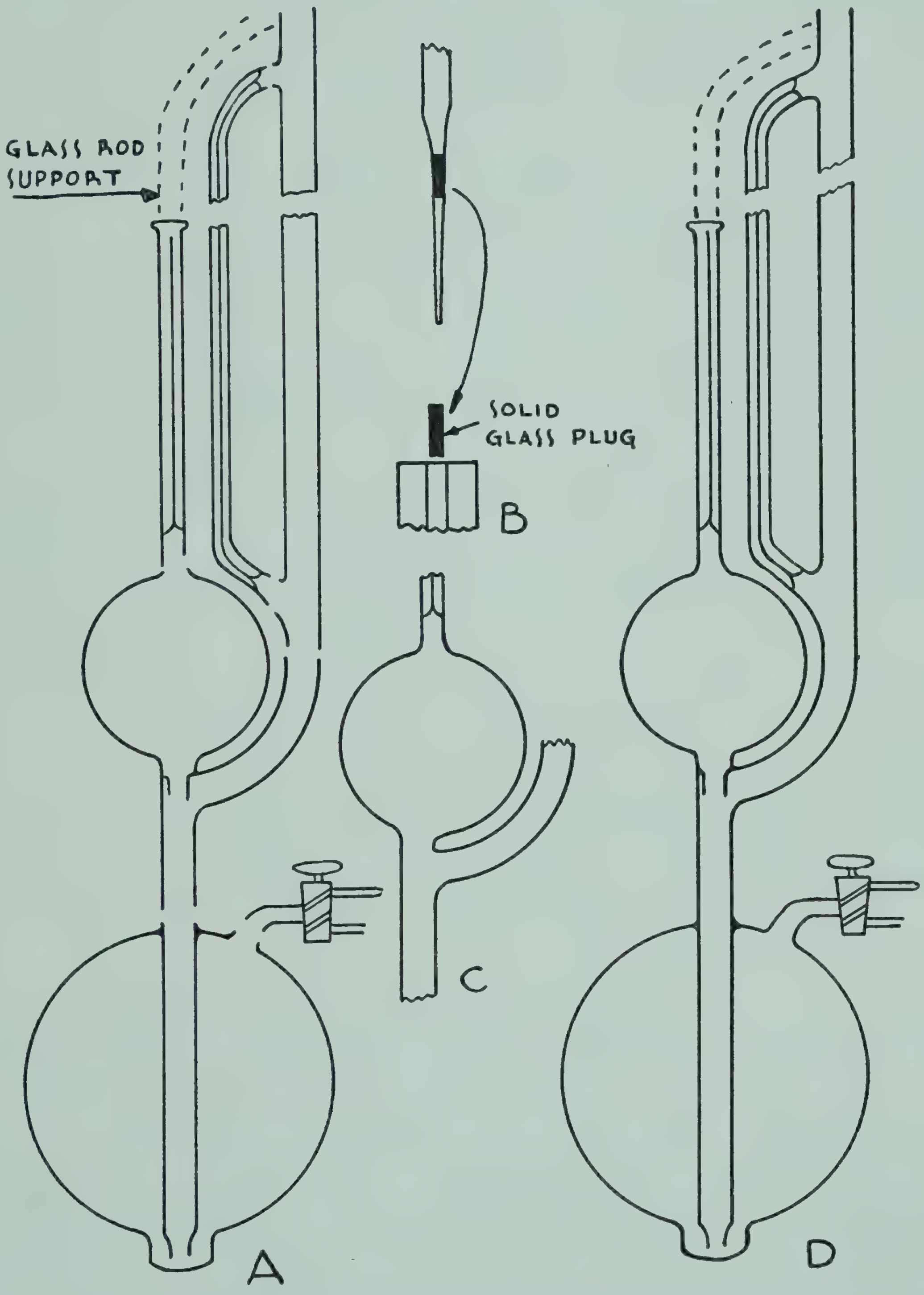


Figure 22. Assembling a McLeod gage.

that the bore of the capillary tubing is not distorted. The end of the capillary is then closed by means of a glass plug, as shown in Figure 22B. Again care should be taken to avoid distorting the capillary bore in making this seal. The capillary is then ready to be sealed to the flask. For highly accurate McLeod gages the ring seal shown at the base of the calibrated flask should be made. (See Figure 22D.) This acts as a cutoff as the level of the mercury is raised. If this accuracy is not necessary the McLeod gage may be made as shown in Figure 22C. In either case the side arm should be sealed so that the mercury will completely fill the tube and not trap air bubbles as it rises.

Calibrating. In calibrating McLeod gages there are two quantities which must be known, one, the volume of the measuring bulb, and two, the volume per unit length of the capillary tubing being used. The first quantity should be determined by weighing the bulb after the capillary and side arm have been sealed in place (Figure 22C). The bulb is then filled with distilled water and again weighed. The temperature of the water should be taken, and with this information the difference in weight between the full and empty bulb will give the volume in cubic centimeters. Data for the second known quantity were secured by weighing the pellet of mercury used in testing the accuracy of the I.D. of the capillary tubing used. Since mercury has a density of 13.6 g. per cubic centimeter at room temperature, the volume of the capillary is expressed as:

$$\text{c.c. per mm.} = \frac{\text{weight of mercury in grams}}{13.6 \times \text{length of the mercury pellet}}.$$

Two different procedures are generally used in reading McLeod gages. In one the mercury is brought to a predetermined mark on the closed end of the capillary, and the pressure is read on the open capillary tube. This method leads to a uniform scale of pressure on the open capillary. For this calibration we have:

V = The volume of the bulb and capillary.
A = Volume of 1 mm. length of closed capillary.
D = Distance in mm. of the selected zero below the closed end of capillary.

H = Difference in height in mm. of mercury between the two capillaries.

or $P = \dfrac{ADH}{V}$.

The second method is always to bring the mercury in the open to be level with the top of the closed section. In which case,

$$P = \frac{AH^2}{V}.$$

This method leads to a nonuniform scale of pressures in the closed capillary, the scale being progressively more open as the pressure is lowered.

A third procedure, which is actually a variation of method No. 1, is "direct calibration." McLeod gages may be read to pressures of 1×10^{-2}, 1×10^{-3}, 1×10^{-4}, and 1×10^{-5} by this method. The procedure is as follows: The flask and the capillary are completely filled with mercury or water and weighed on a reasonably accurate balance. This gives the total volume, which is then broken down as follows:

500.0 g. = total volume
50.0 g. = 1×10^{-1} mm.
5.0 g. = 1×10^{-2} mm.
0.5 g. = 1×10^{-3} mm.
0.05 g. = 1×10^{-4} mm.
0.005 g. = 1×10^{-5} mm.

The amount of mercury calculated for the volume 1×10^{-5} is carefully weighed and inserted into the closed capillary and the glass tapped until the mercury rests on the squared end of the capillary plug. A scratch is then made on the glass at the exact top of the thread of mercury and the additional mercury necessary to complete the weight for the 1×10^{-4} calibration is weighed and inserted in the capillary. Any air bubbles trapped between the two threads of mercury may be removed by tapping on the side of the capillary. A second scratch or calibration mark is made at the top of the mercury level and step two is repeated again, with enough mercury weighed this time to complete the total weight required for the 10^{-3} calibration. The procedure is then repeated once more for the 10^{-2} mark.

Reading pressures on McLeod gages calibrated in this fashion is quite convenient. The mercury is raised to the desired calibration mark and the pressure is the difference in height between the two columns of mercury multiplied by the 10^{-3}, 10^{-4} or whichever mark is used.

Note: Small amounts of mercury can be weighed accurately

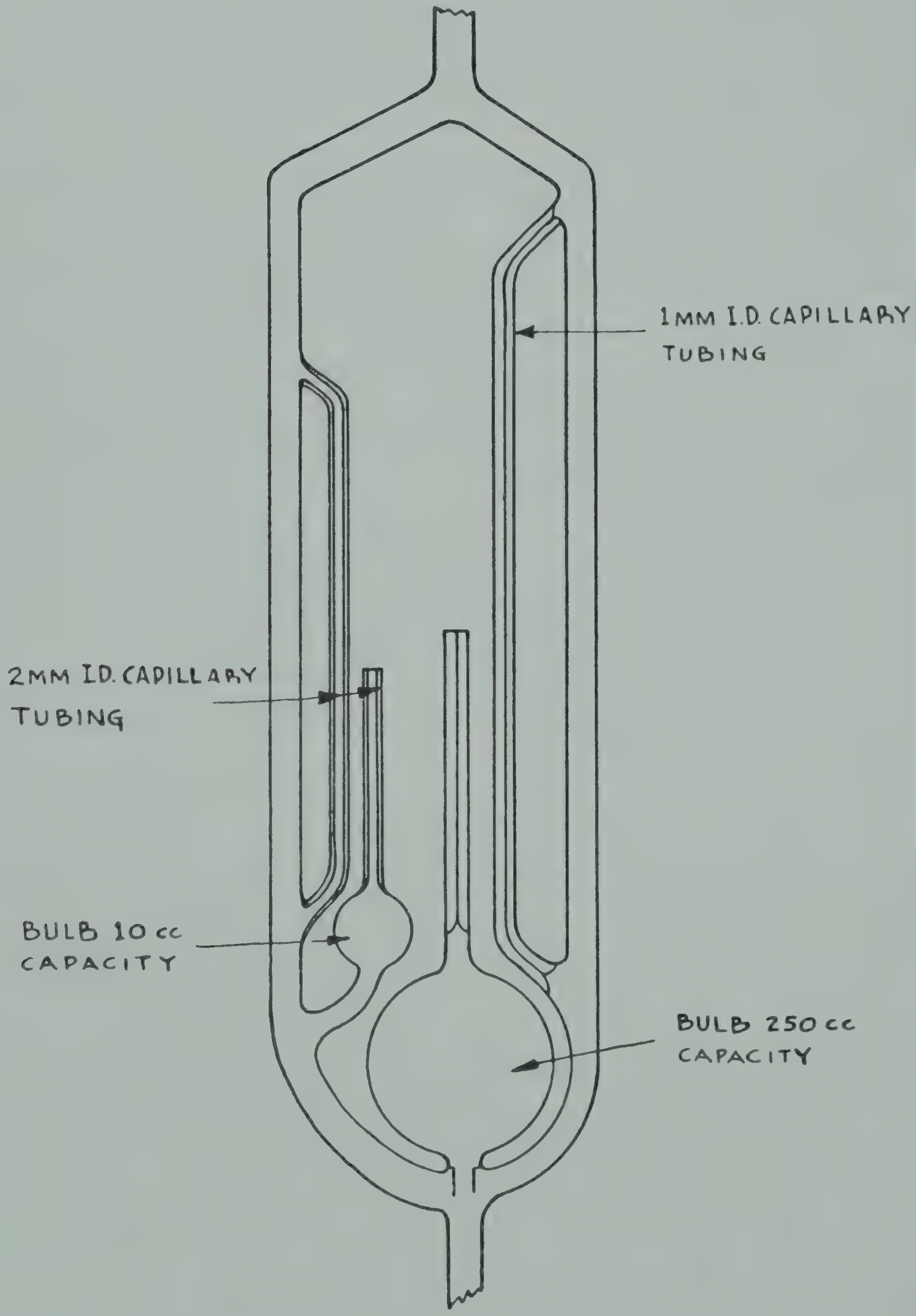

1MM I.D. CAPILLARY TUBING
2MM I.D. CAPILLARY TUBING
BULB 10 cc CAPACITY
BULB 250 cc CAPACITY

Figure 23A. Multiple McLeod gage.

by means of a medicine dropper with a tip which has been drawn to a fine capillary 2 or 3 in. long.

Multiple McLeod Gages. It is possible to extend the pressure range at which a McLeod gage can be read by making two separate gages, both of which are filled from the same mercury reservoir, as shown in Figure 23A. Calibration of each bulb of a multiple gage of this type is carried out as for two separate gages. Figure 23B shows another design for a multiple McLeod gage. This gage has four linear scales and one quadratic scale, giving

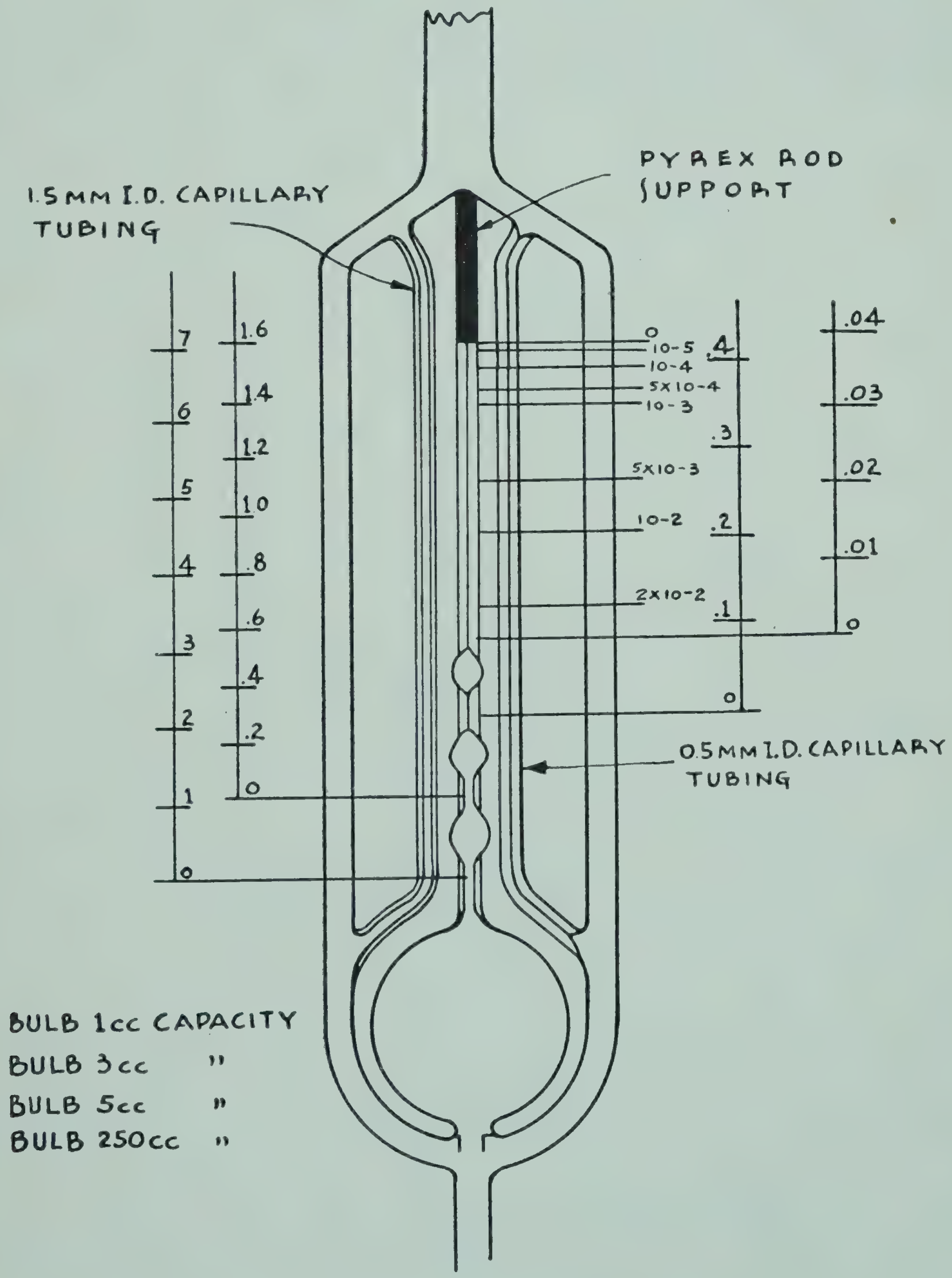

1.5 MM I.D. CAPILLARY TUBING
PYREX ROD SUPPORT
7
6
5
4
3
2
1
0
1.6
1.4
1.2
1.0
.8
.6
.4
.2
0
0
10-5
10-4
5x10-4
10-3
5x10-3
10-2
2x10-2
.4
.3
.2
.1
0
.04
.03
.02
.01
0
0.5 MM I.D. CAPILLARY TUBING
BULB 1cc CAPACITY
BULB 3cc "
BULB 5cc "
BULB 250cc "

Fig. 23B. Multiple McLeod gage.

a pressure measurement over a range from 10^{-5} to 7 mm. of mercury.

High-Pressure (10 to 1) McLeod Gage. These gages may be used to read pressure from 0.1 mm. mercury to as much as 10 mm. mercury, depending on the height of the side arm. Calibration of these gages follows the procedure outlined above. After the weight of mercury necessary to completely fill the bulb up to the side arm is obtained, 1/11 of this amount is weighed out and inserted in the standard-wall tubing above the bulb, and the calibration mark carefully made. The McLeod is read by raising the mercury to this mark in the closed end and the pressure is the difference in levels between the two columns of mercury divided

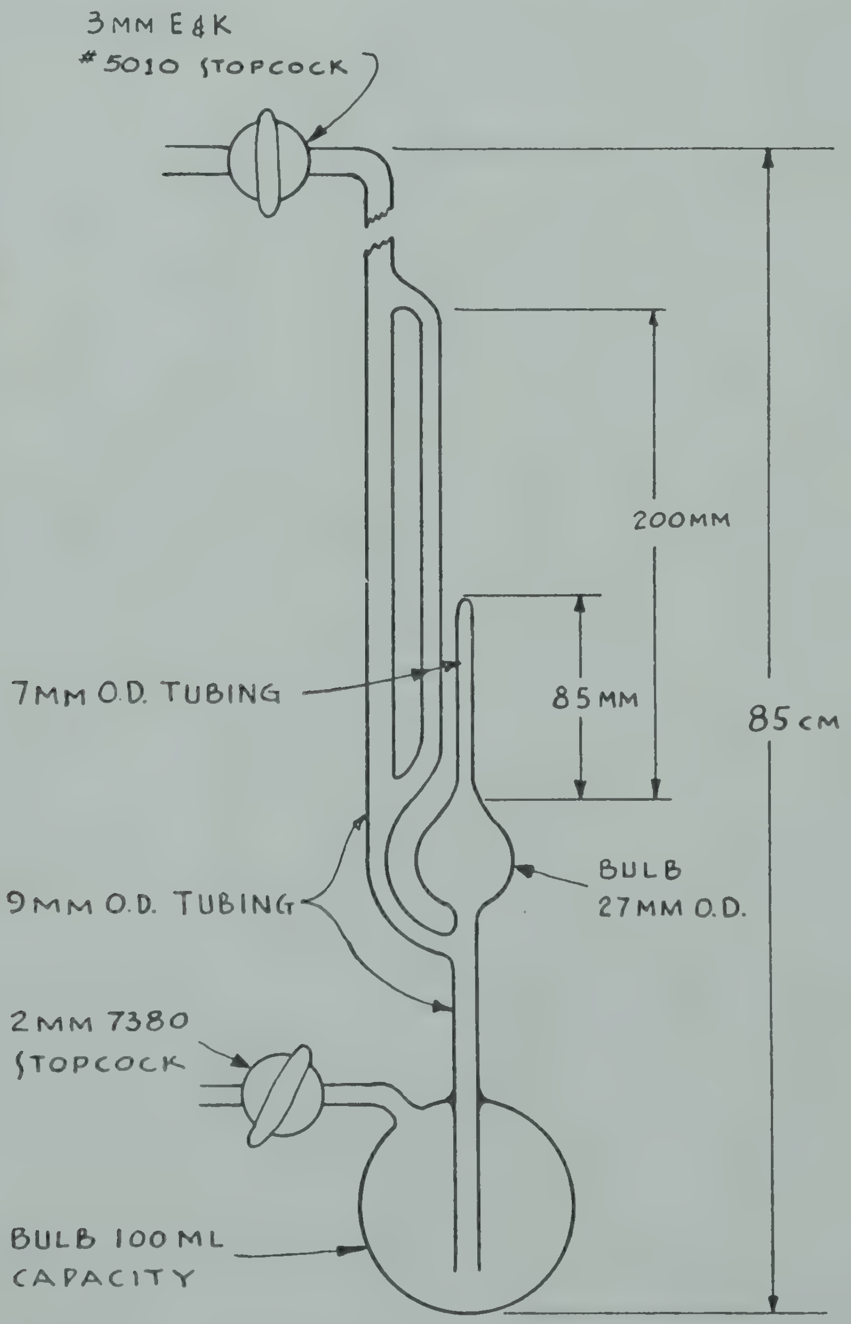

3MM E&K
#5010 STOPCOCK
200MM
85MM
7MM O.D. TUBING
85 CM
BULB
27MM O.D.
9MM O.D. TUBING
2MM 7380
STOPCOCK
BULB 100ML
CAPACITY

Figure 24. High-pressure (10 to 1) McLeod gage.

by ten. Thus a difference in the mercury levels of 1 mm. would mean a pressure of 0.1 mm. (See Figure 24.)

Installation. A a precautionary measure, McLeod gages should extend longer than 760 mm. to prevent mercury from being spread throughout the vacuum system should a stopcock on the reservoir side of the gage develop a leak. The return trap shown in Figure 25 is also a useful safety device to prevent flooding the system with mercury in case of an accident.

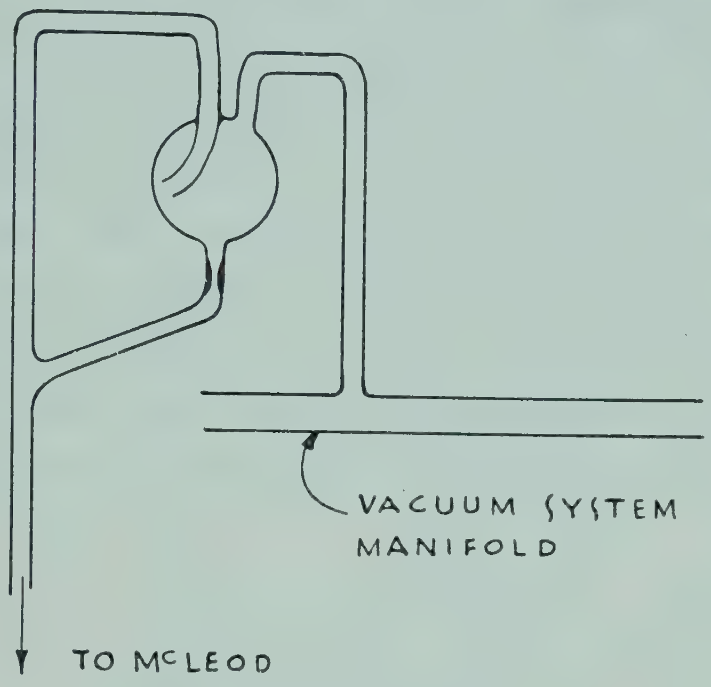


Figure 25. Mercury trap for McLeod gage.

It is desirable that some support be devised for the bulb of the McLeod gage, and this is especially true if a 500-ml. bulb is used. A method of mounting the vacuum system to a large piece of ¾-in. plywood, which has been braced at the edges with a 1-in. angle iron frame, has been used with considerable success by the author even for vacuum systems which were portable and moved from location to location. With this type of mounting it is quite easy to make a support for the calibrated bulb out of a ring support which has a segment of the ring removed so that it can be put in place under the bulb. A piece of soft rubber should be forced over the two pieces of iron ring to make a softer cushion for the bulb. In addition the section of glass tubing between the mercury reservoir and the calibrated bulb should be heated to the softening point after the mercury reservoir is filled. This will remove any strains and stress set up in the glass by the filling and subsequent settling of the mercury reservoir. When a wooden mounting is used for a vacuum system, the glass parts should be mounted 3 to 4 in. away from the wood to allow for repairs and alterations to be made on the system. Stationary vacuum systems may be mounted on iron bar racks with the use of the same clamping arrangement as for wood.

Tilting McLeod Gages. A swinging or tilting type of McLeod gage was described by Flosdorf.[24] This type of gage can be read rapidly and is accurate within the limits of its range. In use a gage of this type is ordinarily supported in the horizontal position, in which position it is evacuated. When the gage is tilted to one of the two other positions, which are in the vertical plane and 180° apart, it can measure the pressure. The gage consists of a central expanded part to which are sealed, on one side, one arm in the form of a U-tube, and another arm, on the other side, which functions as a McLeod gage (see Figure 26). More elaborate variations of this type of gage have been described by Moser.[25]

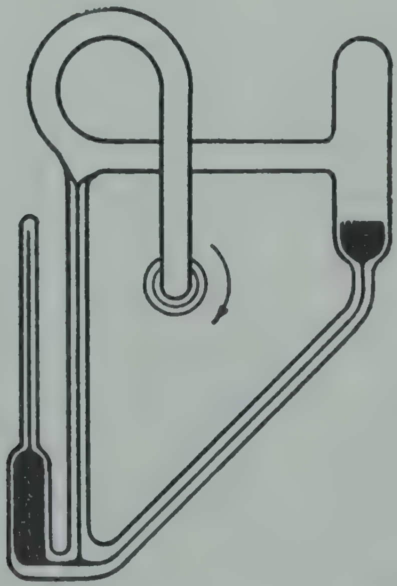


Figure 26. Tilt type McLeod gage. (From A. Weissberger, *Technique of Organic Chemistry*, Vol. IV.)

Bourdon Gages

Deformed Bulb Type Bourdon Gage. This type of gage is shown in Figure 27. This gage depends on the thinness of its walls and its semicircular shape for its sensitivity. This bulb is made as follows: A section of 9 mm. O.D. tubing is drawn into a point with thin walls. When this has cooled sufficiently the section just past the shoulder of the main tube is reheated and blown into a thin-walled bulb. This time it is not necessary to blow a symmetrical bulb. In fact, a thin, off-center bulb is to be preferred (Figure 27). A bushy oxygen flame is then directed at the flat side of the bulb and the glass drawn in by a light sucking action on the blow tube until the bulb looks like that pictured in Figure 27A. In use, these gages are sealed in an envelope as shown in Figure 27B, with a pointer sealed to the top to act as a zero point. A tip of dark-colored glass on each pointer makes reading the gage easier.

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[24] Flosdorf, E. W., *Ind. Eng. Chem., Anal. Ed.*, **17**, 198 (1945).

[25] Moser, H., *Physik. Z.* **36**, 1 (1935).

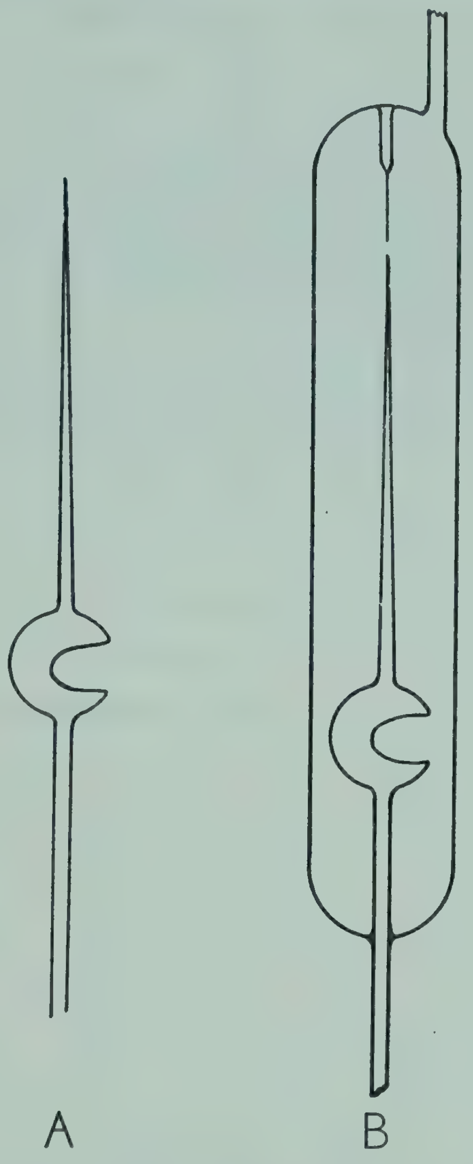

Figure 27. Bourdon gage.

Bourdon gages are used as null-point indicators in which one side with an unknown pressure is balanced against a known pressure. These gages may be made with sensitivities of

0.01 mm. Hg or better, but the more sensitive they are the more fragile they are and liable to breakage. In use a Bourdon gage is mounted in a vertical position with one leg of a mercury U-tube manometer attached to the tube, which is ring sealed to the gage. A change of pressure inside the glass envelope will register in a change of position of the gage pointer. Zero setting can be achieved again by raising or lowering the mercury in the U-tube manometer, and the pressure needed to achieve equilibrium may be read on the manometer.

A modified Bourdon gage was designed by Foord,[26] who attached a thin glass fiber with a mirror to the glass bulb in an ingenious arrangement. Any movement of the pointer in turn moved the glass fiber and mirror and the deflection was registered on a scale mounted 1 meter from the gage. According to Foord a gage which normally would fracture at 200 mm. of mercury could be made to withstand a pressure difference of 1 atmosphere when mounted according to his design. The loss in sensitivity is compensated for, by the multiplication of deflection by the lever system used and by the mounting of the scale away from the gage. Gages with a deflection of 0.54 mm. per millimeter of mercury using a pointer 20 cm. long were reported.

Bourdon Spiral Gage. A Bourdon spiral gage shown in Figure 28B also depends on the mechanical deformation of a thin-wall tube for indicating pressure changes. The spiral consists of a flattened thin-walled glass tube bent in the form of a spiral. Quartz is customarily used for these spirals because of its greater mechanical strength; however, York[27] reported on a method of making these spirals from ordinary commercial glasses. Figure 28A shows a machine built in the University of California shop for winding these coils. The machine has been built with a self-feeding gear with the same pitch as the tapered carbon mandrel, so that in use the section of the mandrel where the coil is being formed is always in the same position under the metal shield and therefore in the same position in regard to the gas-oxygen flame used for heating the glass.

[26] Foord, *J. Sci. Inst.*, **22**, 196 (1945).

[27] York, S. G., *J. Sci. Instruments*, **22**, 196 (1945).

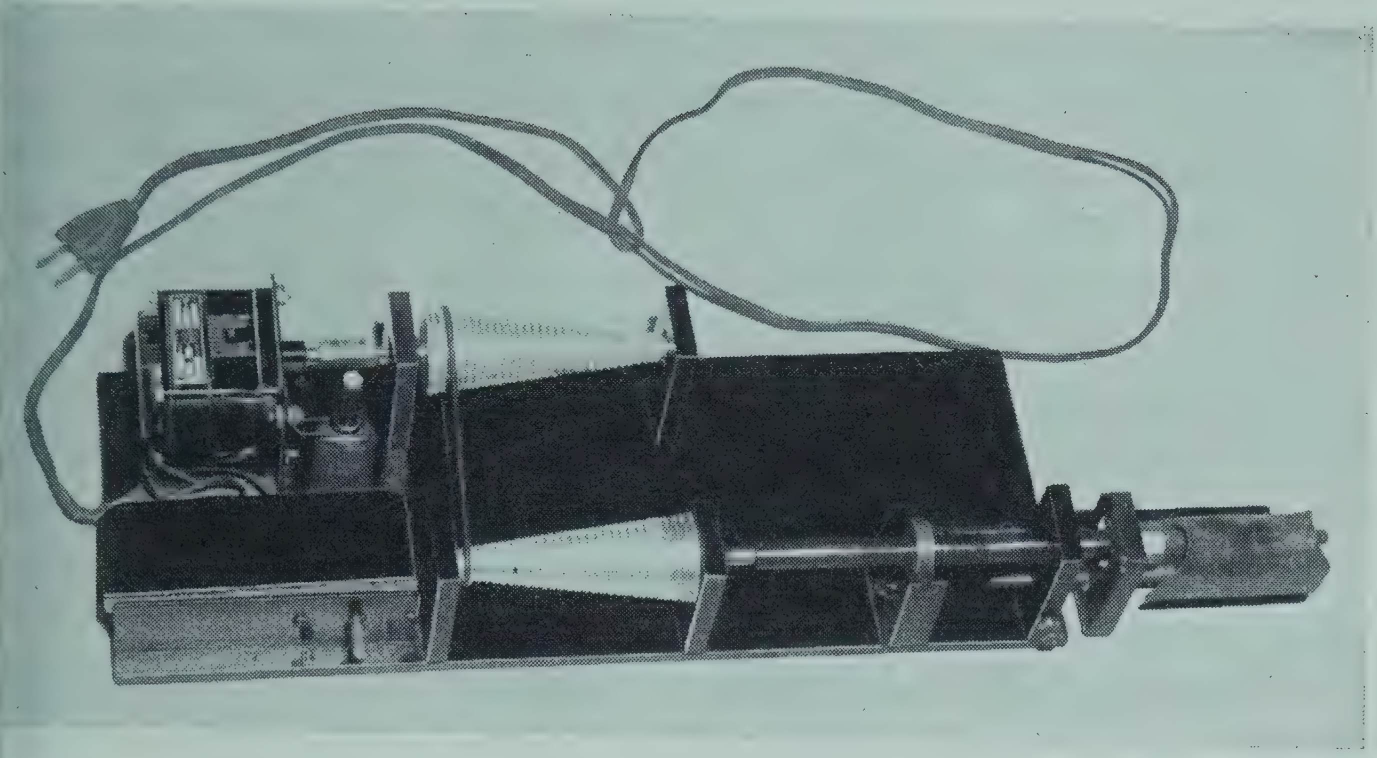

Figure 28A. Machine for winding spiral for Bourdon gages.

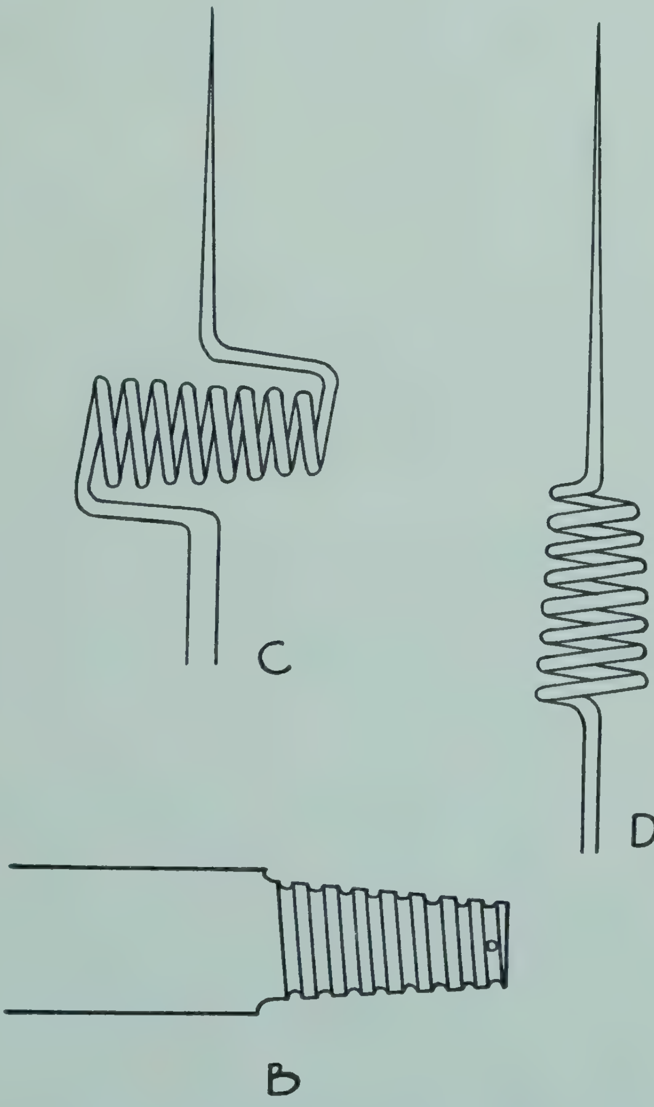

Figure 28B–D. Bourdon spiral gages and form for winding.

The motor is reversible for bringing the mandrel back to the starting position at the completion of each coil. This has the added advantage of making it possible to unwind each coil by merely breaking off the tip which was inserted through the carbon mandrel and holding the coil stationary as the mandrel reverses itself.

It is important that the glass be heated by playing a flame on the metal shield. Glass thin enough to have the required sensitivity will collapse if a flame touches it directly. The author found that satisfactory coils could be wound more easily with uranium glass #3321, because of its lower working temperature, than with Pyrex #7740.

In preparing the thin glass tubing for these coils a section of 8-mm. tubing should be heated and drawn out to a diameter of about 1.25 mm. A 12-in. length of this tubing is then wound into the coil. When the coil is finished the large end should be heated and blown into a bulb of more nearly standard-wall thickness, and this in turn is sealed to a piece of standard-wall tubing preparatory to being ring sealed into the enclosed glass envelopes.

The coil may be mounted either vertically or horizontally, as shown in Figures 28C and D.

Pirani Gages

A Pirani gage is a continuously indicating electric gage which depends on the change in resistance of a heated filament with the change in thermal conduction at various pressures of the gas molecules in the gage. At low pressures, when the mean free path of the gas molecules is of the order of magnitude of the diameter of the gage, the heat transfer is proportional to the pressure. Pirani gages must be calibrated against a McLeod gage, and the calibration varies with each gas used.

Commercial Pirani gages are available[28,29] and are effective through a pressure range of 10^{-1} to 10^{-4} mm. Pirani gages are often used on a high-vacuum system in conjunction with an ionization gage to indicate when the pressure at which it is safe to turn on the ionization gage filament has been reached.

[28] Distillation Products Industries, Rochester, New York.

[29] Continental Electric Co., Geneva, Illinois.

Ionization Gages

An ionization gage depends on the current-carrying capacity of the gas molecules in the gage. When the gage is free of gas, the grid current is extremely small, but when gas is present the gas molecules are ionized as a result of collisions with the electron stream. The positive ions formed drift to the negative plate. Thus the grid current can be used to measure the pressure. Distillation Products Industries[28] make several models. One is the DPM-36 for pressure range of 10^{-3} to 10^{-5} mm. Model DPA-38, for pressures ranging from 10^{-3} to 10^{-9} mm., is designed to automatically shut the filament off whenever the pressure rises 1.2 times full-scale deflection. Ionization gage tubes must be degassed each time the system is opened to the atmosphere. In most tubes a method of degassing the metal parts is supplied; however, the glass envelope must be degassed by means of a gas-air flame. When the glass envelope is made of Nonex glass, extreme care must be taken in the flame degassing and the flame should be gas with no compressed air mixed with it.

Knudsen Gage

A Knudsen[30] gage is the only gage other than a McLeod gage which will read pressure directly. These gages are preferable to McLeod gages for systems in which contamination by mercury vapor must be avoided. Complete instructions for making a Knudsen gage as designed by DuMond have been published.[31]

EVACUATION OF GLASS VESSELS

The process for evacuating and baking out flasks has been suggested by the results of experiments by Langmuir,[32,33] who concluded that to remove water vapor efficiently from the walls of glass vessels the heating during evacuation should be carried out in two or more stages of decreasing temperature. This is in accordance with similar observations made by Sherwood,[34] who stated that gas evolution at temperatures below 500° C. practically ceases after one-half hour, but that the evolution of water vapor at higher temperatures continues indefinitely no matter how long the heating periods. Apparently glass suffers actual chemical decomposition at higher temperatures.

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[30] Knudsen, M., *Ann. Physik*, **28**, 75 (1909).

[31] Du Mond, J. W. M., and Pickels, W. M. F., *Rev. Sci. Instr.*, **6**, 360 (1936).

[32] Langmuir, *Trans. Am. Inst. Elec. Engrs.* **32**, (1921).

[33] Langmuir, *J. Am. Chem. Soc.*, **38**, 2221 (1916).

According to Dushman[35] the reasons for evacuating and baking a glass vessel at 400° C. after a period at 550° C. are as follows:

At temperatures near the softening range, glass undergoes marked changes and large quantities of gas diffuse from the interior out through the surface. While the gases are diffusing, their concentration at the surface is high. If the glass is cooled rapidly from these high temperatures and sealed off, much of the gas at the surface is retained by the glass and given off slowly after sealing off. Although there is small diffusion of gas from the interior of glass at 400° C., gas from the surface can be effectively removed by baking a short time at this lower temperature. It is not sufficient to remove only the gases absorbed on the surface because, after sealing off, gases from the interior will diffuse slowly into the evacuated space.

Evacuation of Unsilvered Vessels

When unsilvered vessels are to be evacuated it is advisable to clean them with either cleaning solution or a rinse with 0.7 molal hydrofluoric acid. Following this wash, several rinses with distilled water and a rinse with ammonium hydroxide are in order, along with a final methanol rinse before drying the vessel preparatory to its being sealed on the vacuum system. A rinse with methanol after the distilled water rinses will materially reduce the pumping time necessary to dry the vessel before sealing it on to the high-vacuum system. When a mechanical pump is used for prepumping, it should be well protected by means of a dry ice trap between the vessel and the pump.

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[34] Sherwood, R. G., *J. Am. Chem. Soc.*, **40**, 1645 (1918); *Phys. Rev.*, **12**, 44 (1918).

[35] Dushman, S., *Scientific Foundations of Vacuum Technique*, p. 511 Wiley, New York, 1949.

It is desirable that the oven be designed with a small door or some other suitable arrangement for opening the oven near the tubulation in order to facilitate sealing off the evacuated vessel while it is hot. In a Lyddon oven, designed and built for the University of California at Los Angeles especially for evacuating distillation columns, the columns are supported by an angle-iron frame within the oven, and the vacuum line enters the oven through the bottom. There are four vacuum outlets, allowing four columns or vessels to be evacuated at one time.

Glass Baking during Evacuation

The column or jacket to be evacuated is supported in the oven and the tubulation is sealed to the high-vacuum system. The fore pump is started and allowed to pump until a pressure of 1×10^{-3} mm. is reached. When this pressure has been reached, the mercury diffusion pumps are turned on and a Dewar filled with liquid nitrogen is placed over the trap between the system and the silvered vessel to prevent the diffusion of mercury into the column and its amalgamation with the silver. When the pressure has reached a sticking vacuum with the McLeod gage, or 1×10^{-6} mm. on an ion gage, the furnace heater is turned on. The temperature can be increased rapidly when unsilvered vessels are being evacuated. With silvered jackets, the temperature rise should not exceed 1.2° C. per minute, taking about 8 hours to go from room temperature to 550° C. A schedule found satisfactory by the author for use in evacuating one or more large jacketed columns at one time is as follows:

1. The column or columns are sealed to the high-vacuum leads and the fore pump started. When the prescribed 1×10^{-3} mm. pressure reading has been reached, liquid nitrogen is placed around the system trap, and the diffusion pump started and allowed to run overnight.

2. A pressure of 1×10^{-7} mm. is obtained by morning, at which time the oven controls are set for 110° C. and the heaters turned on. Temperature is raised in steps of about 115° C. during

the day ($2\frac{1}{2}$ to 3-hour intervals) until 450° C. is reached, at which temperature the oven is held during the second night.

3. In the morning the pressure should again read 1×10^{-7} mm. The oven temperature is then raised to 550° C. and held for 1 hour, after which it is allowed to drop to 400° C. and again held for 1 hour, or until the pressure reads 1×10^{-7} mm.

4. At this point the lower section of oven door is opened and the column sealed off from the vacuum system.

Sealing Off Evacuated Vessels

Care must be taken in sealing off evacuated apparatus to allow the gases liberated during sealing to be pumped off.

Constrictions in glass vessels at the point where they are to be sealed off after exhaust should not be too thick-walled; otherwise a large body of glass will have to be heated during the sealing off, causing the liberation of a large volume of gas. Furthermore, the constriction should be heated by torch until almost melting and the pump allowed to exhaust the gas thus liberated for about 2 minutes, after which the sealing off should be performed as rapidly as possible without heating the glass any more than absolutely necessary. The actual time allowed for removing the liberated gases will depend on the size of the vacuum system and the speed of the pump. If an ion gage is used in conjunction with the vacuum system, an accurate check is available.

Effect of High Temperature on Silvered Surfaces

Heating a silvered surface to a high temperature in a vacuum frequently spoils the appearance. The silver becomes gray and semitransparent and not infrequently scales off the glass. A careful check will tell whether the scaling-off is from liquids contained in a striation (see page 192) or from some other cause. According to Scott, Cook, and Brichwilde[36] the damaged silver surface is not due to any specific effect of the high temperature but comes primarily from raising the temperature too rapidly.

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[36] Scott, R. B., Cook, J. W., and Brichwilde, F. G., *Bur. Standards J. Research,* **7**, 936 (1921).

This can be prevented, to a large extent, by cleaning as described (see page 194); this is not always necessary if the temperature is raised slowly enough. Before baking out, the glass side of silver on an etched flask is duller than that of an unetched flask, although the appearance improves during the baking out. Scott found no difference between the insulating qualities of etched and unetched flasks. However, tests showed that heating to a high temperature improves the reflectivity of the vacuum side of a silvered surface for visible light and that the higher the temperature, the higher the reflectivity.

Notes on Evacuation Practices

When glass apparatus is sealed to the vacuum system preparatory to being pumped or when repairs are being made to the system, blowing should be through a drying tube.

2. At the completion of each evacuation the system should be filled with nitrogen at atmospheric pressure. If this is done the system will remain dry and the ion gage will need a minimum of degassing at the beginning of the next evacuation run.

3. When nitrogen is being introduced into the system, the McLeod gage is ideal for indicating when the system is at atmospheric pressure if air is allowed to enter the mercury reservoir as the nitrogen is passed into the vacuum system. The pressure of nitrogen in the system should be allowed to build up slightly faster than the air in the reservoir in order to force a column of mercury up the McLeod gage. When the stopcock on the mercury reservoir is wide open and the column of mercury in the McLeod drops to the level of the mercury in the reservoir, the system is at atmospheric pressure.

The stopcock should be turned to the reservoir evacuation position as soon as the system has equilibrated, so that the McLeod gage will be ready the next time the pump is turned on.

DETECTION OF LEAKS

The location and repair of small leaks in a vacuum system is one of the major problems encountered in working at reduced pressures.

Tesla Coil

Many leaks may be found in all-glass systems by means of a Tesla spark coil. The location of the pinhole will be shown by the electric discharge concentrating on and passing through the hole with an intensely bright glow as compared to the discharge spreading over the surface of the glass when there is no leak.

At times it is possible to puncture the glass with too intense a spark. This, however, can be done only when the glass is very thin or when there is a flaw in either the glass or glassblowing seals. Since such a spot would be a potential leak, it should be located and repaired.

Discharge Tube

A leak in a vacuum system which includes metal sections will require a different technique. The discharge tube shown in Figure 20 should be connected to the vacuum system and the system evacuated until the characteristic pink discharge is formed. The suspected part is then painted with alcohol or acetone and a change in color of the discharge from pink to blue is watched for. Webster[37] recommends that the discharge tube be placed between the diffusion pump and the backing pump. In this position, if the backing pump is shut off before painting the suspected parts with alcohol or acetone, the backing pressure of the diffusion pump will rise rapidly if there is a leak, resulting in a striking change in the color of the discharge. See Table V for the appearance and color of discharges in gases and vapors at low pressures.

If the leak is very small and the system can be evacuated below the pressure at which a discharge is clearly visible, an ionization gage may be used to indicate the entrance of alcohol or acetone vapors into the system. Manley, Haworth, and Luebke[38] reported the change in the electron emission from the filament of the ionization gage to be more sensitive to these vapors than the ion current. Lawton[39] also reported on the use of an ionization gage as a leak detector, but preferred to use hydrogen or oxygen for searching. These gases have the added advantage over liquids of not temporarily closing the hole.

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[37] Webster, *Rev. Sci. Inst.* **5**, 42 (1934).

[38] Manley, Haworth, and Luebke, *Rev. Sci. Instr.*, **10**, 389 (1939).

[39] Lawton, *Rev. Sci. Instr.*, **11**, 134 (1940).

Leak Detector

A halogen-sensitive leak detector is supplied by Distillation Products Industries[40] which uses Freon 12 as a probe gas. Since the molecular weight of Freon 12 is approximately four times that of air, the operator should start at the bottom of the system and work up when probing for leaks. When the general area of a leak is located, the area should be divided into smaller areas and the probing continued. This leak detector is sensitive to all halogen gases as well as combustible gases and the presence of any of these will result in inaccurate results.

The most sensitive type of leak detector is a mass spectrometer, using helium for the probe gas. Commercial leak detectors are available which are essentially a small mass spectrometer and which are adjusted specifically to detect helium. With these even the smallest leaks may be quickly and accurately located, although the initial cost of this detector is probably prohibitive in most cases.

Testing Tungsten-Glass Seals

A method of detecting leaks around tungsten seals is shown in Figure 29. This method is useful with leaks too small to be located using the change of color of the discharge in organic vapors.

Method of Operation. A good vacuum pump is connected to tube B by arm C. The pressure in tube B can be observed by a discharge from electrode D, and if the pressure in the system does not improve when a good vacuum has been pulled on tube B, it is safe to assume the leak is not in the joint being tested. If, however, the vacuum in the system improves as the vacuum improves in tube B, it is a very good indication that there is a leak in the joint.

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[40] Distillation Products Industries, Rochester, New York.

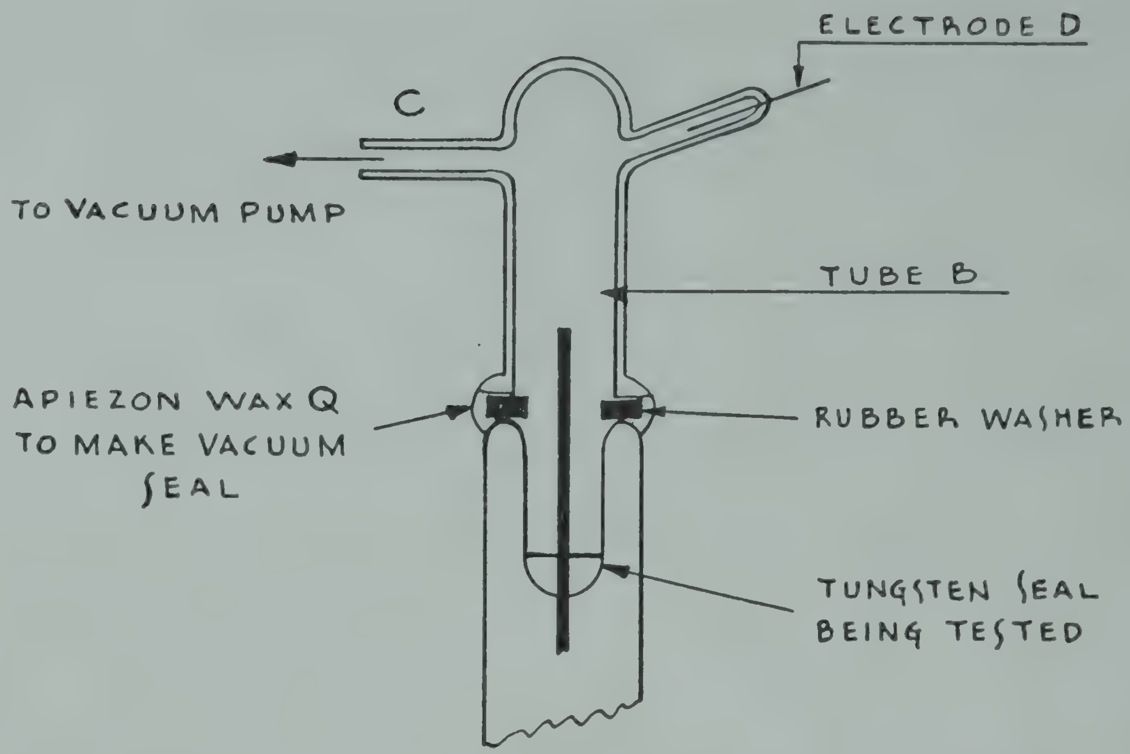
ELECTRODE D
C
TO VACUUM PUMP
TUBE B
APIEZON WAX Q TO MAKE VACUUM SEAL
RUBBER WASHER
TUNGSTEN SEAL BEING TESTED

Figure 29. Device for testing tungsten leads for leaks.

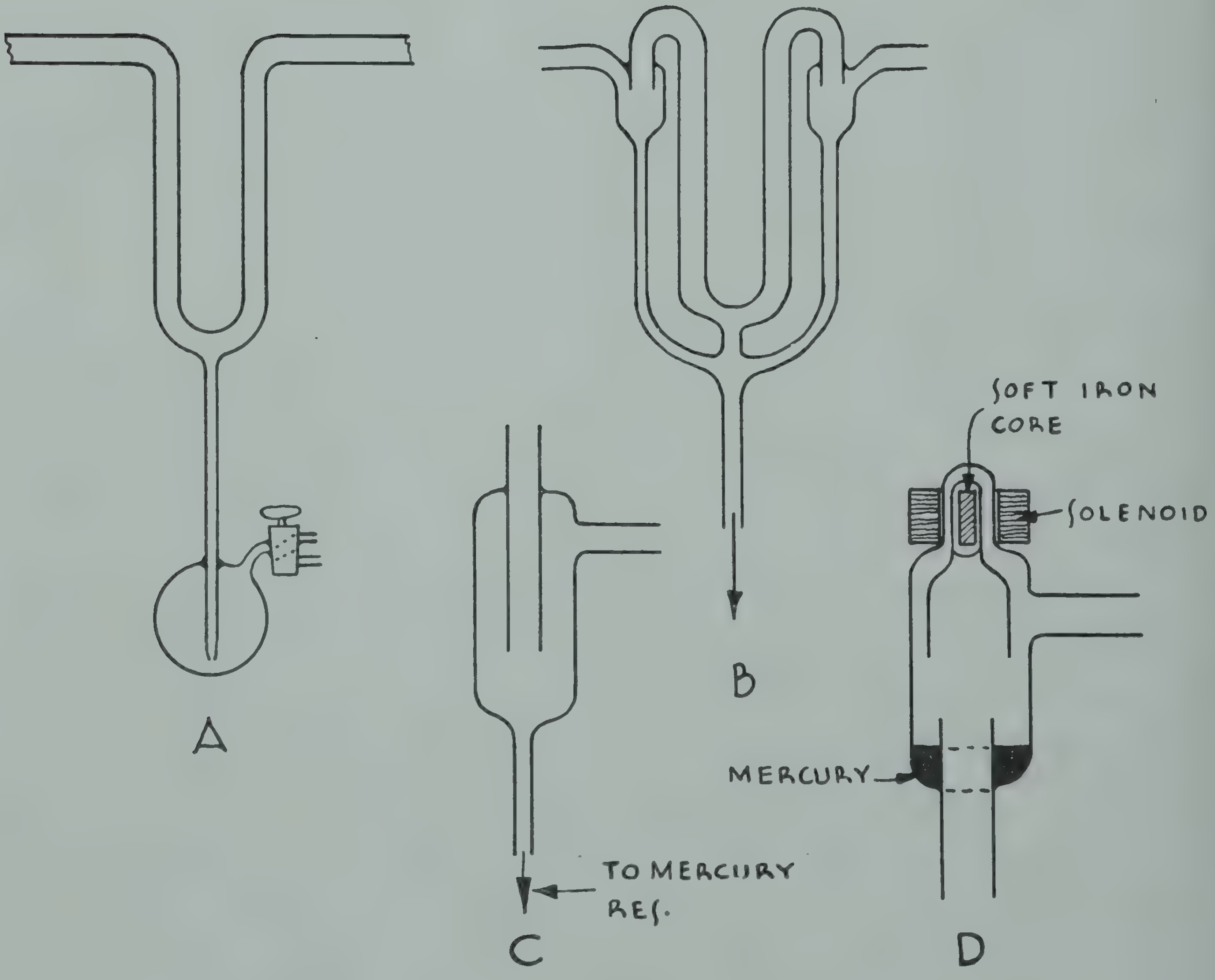
A
B
SOFT IRON CORE
SOLENOID
MERCURY
TO MERCURY RES.
C
D

Figure 30. Cutoffs for vacuum systems.

CUTOFFS

In searching for leaks in a vacuum system it is often desirable to be able to isolate sections of the system or to isolate the vessel being evacuated from the exhausting system. Glass stopcocks have the disadvantage of introducing vapor from the grease into the system. In place of stopcocks, mercury cutoffs may be used. Figure 30 shows several different types of cutoffs which can be made and which offer a free flow of gas when the cutoff is open. In Figure 30, A, B and C are barometric height cutoffs and may be made of any size tubing desired. Figure 30D is a magnetically controlled cutoff and should not normally be operated at pressure differences exceeding a few millimeters.

TOEPLER PUMPS

Toepler pumps were mentioned very briefly earlier in this chapter and described as a slow means of obtaining a vacuum. However, an automatic Toepler pump as designed by Urry *et al.*[41] eliminates many of the objectionable features of previous designs. According to Urry these pumps have proved useful in the quantitative transfer of gases and volatile liquids for gas analysis, reactions in vacuum line apparatus, analysis for adsorbed gas in solids, and other applications. With this new design the pump is more compact and takes up less space, and because the mercury is better supported there is less danger from breakage. The pump can be made any reasonable size and a pump with a piston volume of 800 c.c. operating with a piston cycle of 35 seconds, can evacuate a 2-liter flask to 15 % of the original gas in 10 minutes.

Pumps up to 200 c.c. piston volume size may be assembled with hand glassblowing operations. Larger sizes require lathe operations. The pump is made from a single length of large-diameter Pyrex tubing which is separated into the upper piston chamber and the reservoir by a large ring seal and dip tube A as shown in Figure 31A. The valves, B and C, at the top of the piston chamber are made of glass with conically ground seats. The float

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[41] Urry, Grant, and Urry, W. H., University of Chicago, private communication.

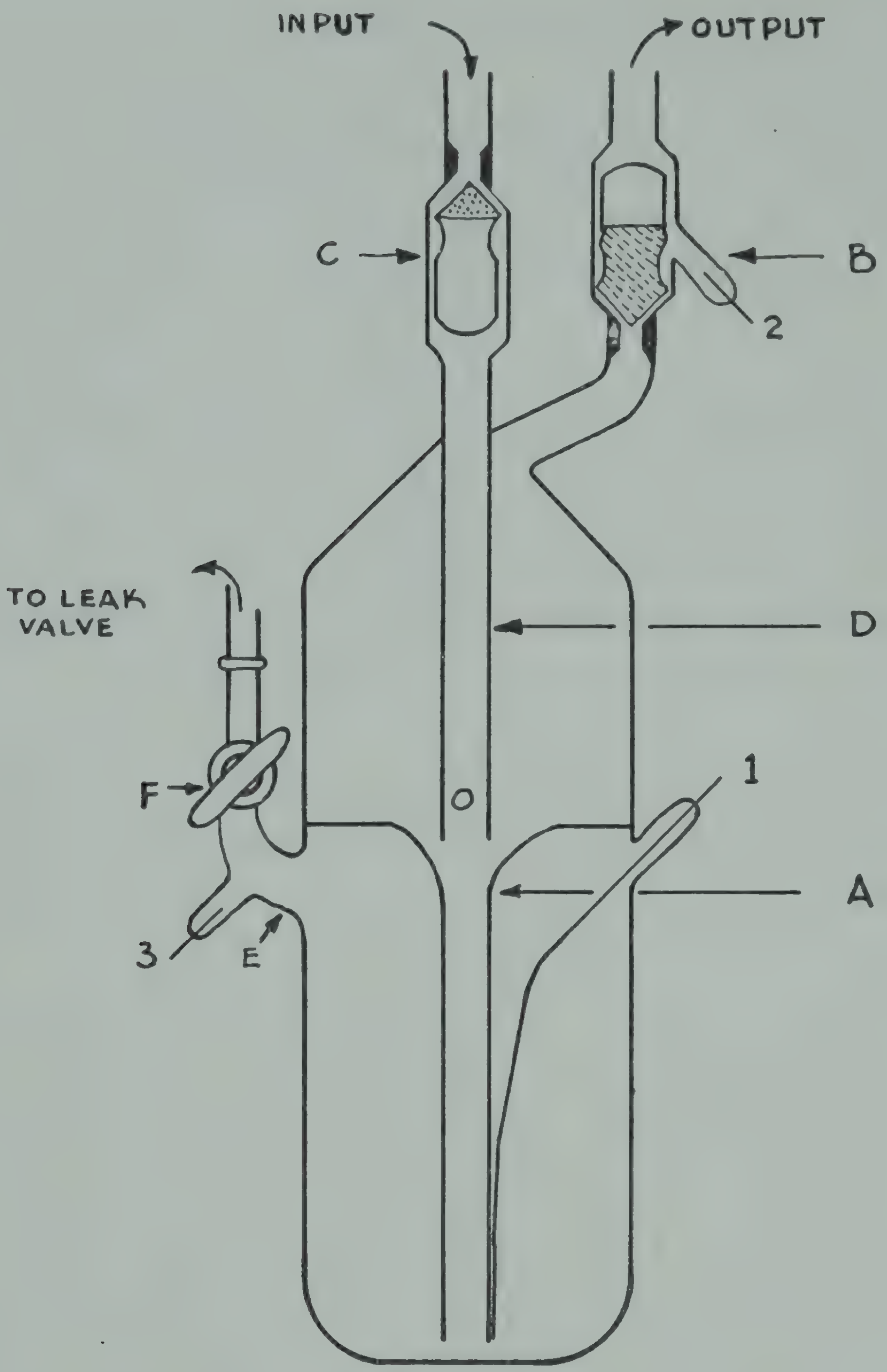

Figure 31A. Automatic Toepler pump.

of the output valve B is partially filled with mercury and that of the input valve C is a simple air float. The input dip tube D, extending to the bottom of the piston chamber, has an opening of 5 mm. diameter 10 mm. from its lower end. The working piston volume is the volume of the piston chamber above the top of this opening. The volumes of the two chambers should be such that the reservoir contains sufficient mercury to fill the piston chamber, dip tubes, and valves, with enough remaining to cover the end of the lower dip tube A. A side arm E and stopcock F are placed at the top of the reservoir chamber near the large ring seal as shown. Tungsten wires are sealed through the glass at 1, 2, and 3.

Pump Operation

The pump is mounted with the reservoir cast in plaster of paris to give support and the reservoir is filled with mercury to the level A. The pump can then be joined to the vacuum apparatus with which it is to be used and the piston chamber and reservoir evacuated. The gas to be pumped is allowed to flow into the piston chamber through the input dip tube D. Air is then allowed to enter the reservoir chamber slowly through the stopcock F near the top of this chamber. This air forces mercury through the lower dip tube and causes mercury to rise in the piston chamber. As the mercury rises it closes the input dip tube D and pushes the gas in the piston chamber past the mercury-filled output valve B. The mercury which rises in the input dip tube is held in check by valve C. When mercury reaches the level of B, electrical contact is established between tungsten wires 1 and 2. This contact closes a relay (Figure 31B) which stops the air entering through F and E (the leak is stopped by relay action), and turns on an oil pump to evacuate the reservoir chamber. The mercury level falls below valve B which closes while its seat is still covered with mercury to insure positive closure. The piston chamber is evacuated on the downstroke and another portion of gas is allowed to enter in small bubbles through the opening in the input dip tube as the mercury level falls. The mercury level falls in the piston and rises in the reservoir until it reaches level A and elec-

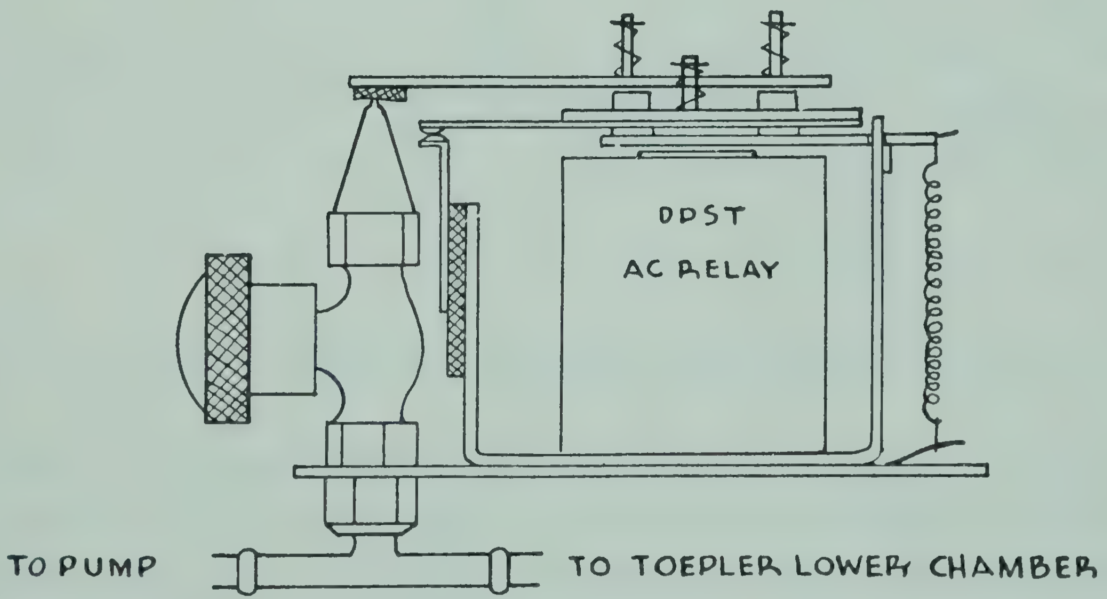

Figure 31B. Relay and leak for automatic Toepler pump.

trical contact is established between tungsten leads 1 and 3. This contact shorts out the relay to turn off the pump and to open the relay leak and the air is allowed to enter the reservoir chamber again. The cycle then automatically repeats itself.

The wiring diagram for the control relay is shown in Figure 31C. Control of the air flow into the lower reservoir chamber can be managed in many ways. Perhaps the simplest is a constant air leak with the oil pump operating against this leak on the downstroke of the pump. When the oil pump is turned off by the relay, the air pressure can build up in the reservoir chamber to cause the upstroke of the piston. Since the size of the air leak must be accurately adjusted to avoid rushing of the mercury piston and consequent damage to the glass valves, it is wise to use a needle valve and to adjust the opening of the valve to obtain a smooth piston stroke. Although the constant leak arrangement is the simplest, some speed is lost when it is used (the leak slows the evacuation of the reservoir, and the upstroke is slowed because the leak must be accommodated to the speed of the oil pump). For the fastest safe operation of the pump it is advisable

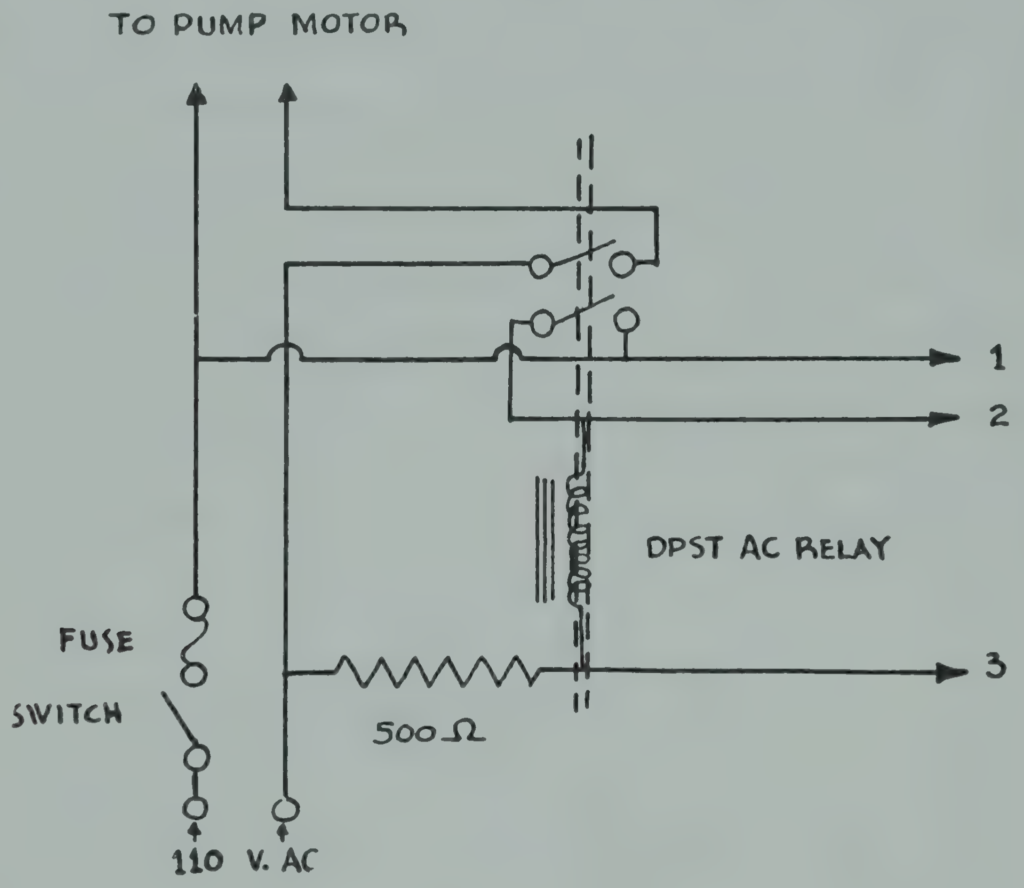

Figure 31C. Electrical diagram for automatic Toepler pump.

to close the air leak when the oil pump is turned on. This can be done with any commercial solenoid used in combination with the needle valve air leak. The solenoid in this case is switched on by the same relay which operates the oil pump. The most convenient scheme, however, has proved to be that shown in Figure 31B. The switching relay, a DPST a.-c. relay (Potter-Bromfield #PR7A), is fitted with a lever arm mounted on the rocker between the contact arms of the relay by means of spring bolts. The end of this lever is fitted with a rubber pad so arranged that when the relay closes, the rubber pad closes the air leak, in this case a Hoke #431 needle valve. The oil pump and Toepler connections are also indicated in Figure 31B.

In the operation of the pump great care must be taken at the beginning of automatic operation to avoid a full rush of air into the reservoir chamber. This causes a violent rush of mercury in the piston chamber which can break it. This rush of mercury can be prevented by placing a constriction in the lower dip tube. This precaution, however, sacrifices the high speed of pumping so desirable in this design. For this reason the extra care necessary with a full flow dip tube is rewarded by a great saving in operating time.

FILLING MANOMETERS

Concentric-Tube Manometer

Concentric-tube manometers can be evacuated by means of a high-vacuum rotary oil pump without the aid of a diffusion pump, providing the rotary pump is in good condition.

Before the manometer is evacuated enough mercury is poured into it to fill the end, leaving only the tip of the inner tube exposed when the manometer is supported at the angle shown in Figure 32. A dry-ice trap should be inserted in the vacuum line between the manometer and the rotary oil pump to protect it from the mercury vapors evolved during the boiling of the mercury.

After the manometer has been evacuated to a vacuum of 10^{-3} mm. or better, the mercury should be boiled to liberate any trapped bubbles of air. The manometer may then be tilted to a vertical position and air allowed to enter, bringing the manometer to atmospheric pressure. If evacuation is complete a column of mercury will fill the inner tube with mercury with no trace of an air bubble.

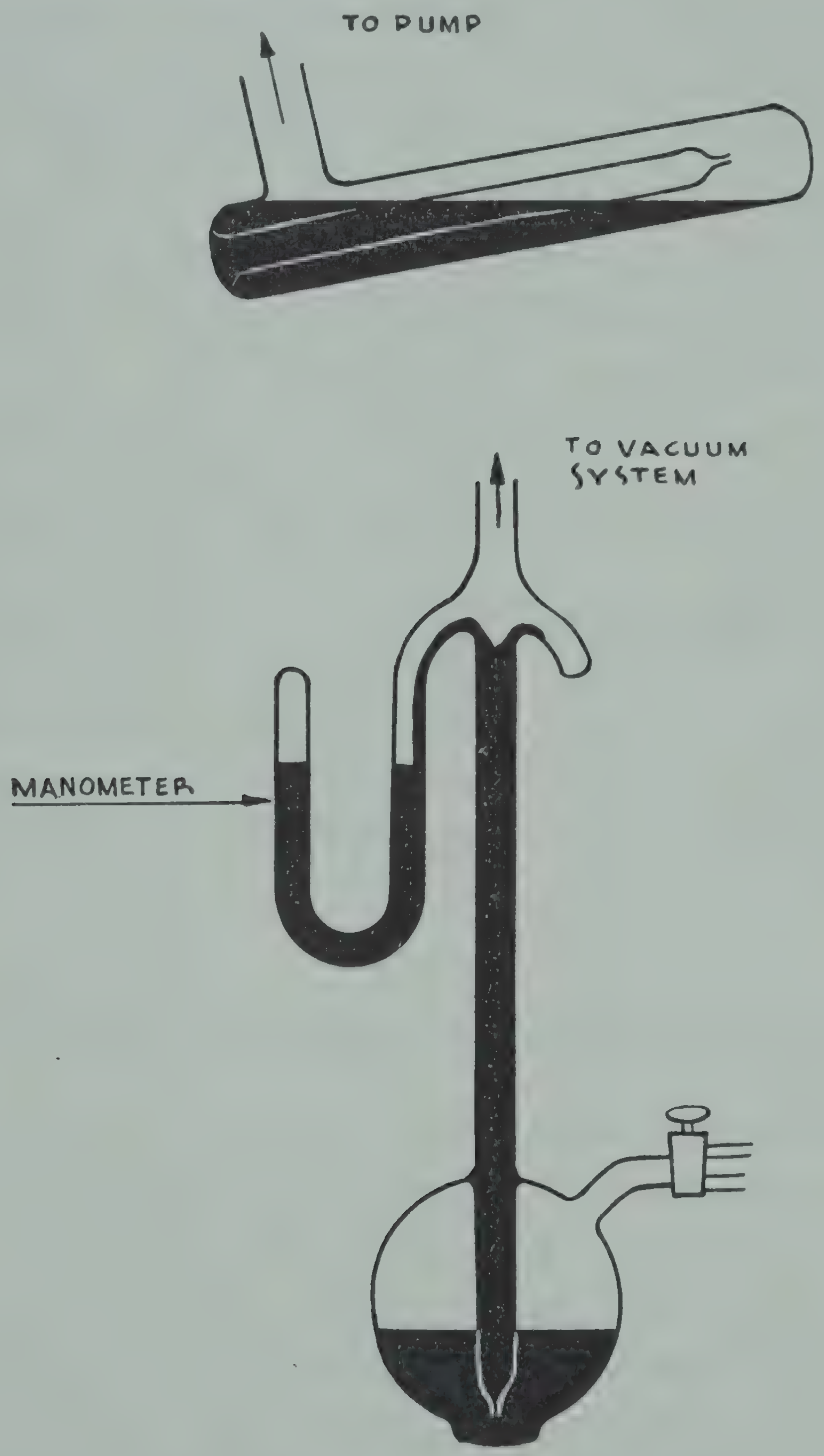

Figure 32. Filling mercury manometers.

Should an air bubble show in the inner tube it will be necessary to empty the inner tube by pumping out the mercury and repeating the complete evacuation process. Special care should be taken to see that there are no vacuum leaks around the rubber connections to the vacuum pump.

Standard U-Type Manometers

This type of manometer is more difficult to fill and usually requires special apparatus. As a rule it is desirable to evacuate U-type manometers by means of an oil rotary pump and a mercury diffusion pumping system. Figure 32 also shows a device which may be sealed to any vacuum system and used for producing manometers of high quality. The flask is the mercury reservoir from which the manometers are filled and should be filled with fresh triple-distilled mercury. After the manometer has been evacuated to a hard vacuum as indicated by a gage on the vacuum system, air should be allowed to enter the mercury reservoir, forcing mercury up to the level, from which it drops into the manometer. The flask may be made with several seal-off tubes for filling more than one manometer at a time.

At no time during the filling process should the manometer walls be flamed to bake them out. Schumacher[42] showed that flaming the glass drove gas from the surface of the glass and resulted in mercury wetting the glass. When this happens the manometer can in time become inaccurate and will have to be replaced.

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[42] Schumacher, E. E., *J. Am. Chem. Soc.*, **45**, 2255 (1923).

XIV

METAL WORKING

A small machine lathe is a very useful piece of equipment in a glassblowing shop, and this is doubly true if the glassworker has a fundamental knowledge of the care and use of such a lathe. Figure 1 shows a standard 9-in. gear change lathe, a model which should be very popular in a glassblowing shop, where few changes in threads and feeds are necessary. Another factor in favor of standard gear change lathes over the quick-change lathes is the initial cost, the standard gear change lathes being much the less expensive of the two.

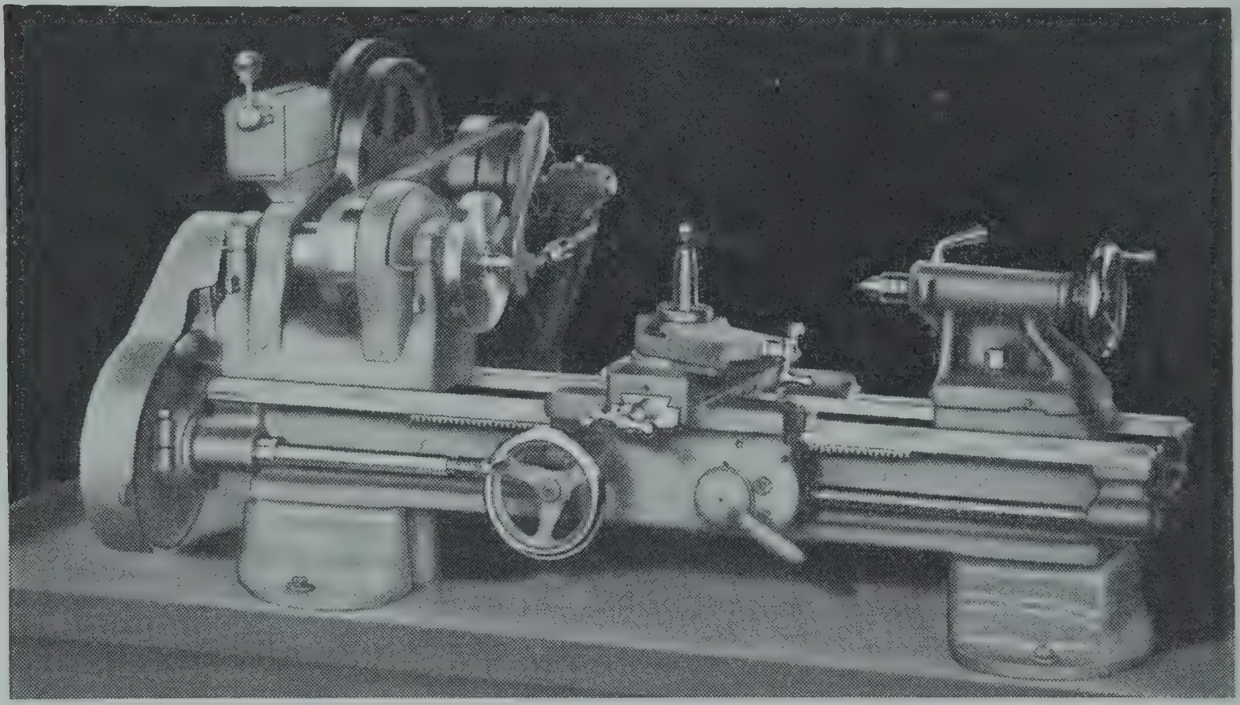


Figure 1. Standard 9 in. machine lathe. (Courtesy of South Bend Lathe Works.)

SETTING UP A MACHINE LATHE

A lathe should be installed very carefully so that all the accuracy built into the lathe is maintained. If a floor model lathe is being installed it should be mounted on a solid flooring, and a concrete base is recommended. Lathes are leveled by placing a shim of hard wood or metal under the legs. The accuracy of the leveling should be checked by means of a precision level at least 12 in. long and sufficiently sensitive that it will show a distinct movement of the bubble when a shim of 0.003 in. is placed under one end. A lathe should be leveled at both the head stock and tail stock. (See Figure 2.) A lathe which is not level will sit unevenly on all four legs and the weight of the lathe will cause the lathe bed to twist. This will result in the headstock's being out of alignment with the V ways of the bed, causing the lathe to turn and bore tapered. It is not possible to do accurate work on a lathe which is not level.

Setting Up a Bench Lathe

Bench lathes should be mounted on a substantial bench which is about 28 in. high and the top should be made of wood at least 2 in. thick. In addition, the bench should be bolted to the floor to prevent shifting and throwing the lathe out of line. Some bench lathes have leveling screws in the right leg which may be used for the final leveling adjustment.

Readjusting Shims

It may be necessary from time to time to readjust the shims under the legs of the lathe to compensate for the settling of the building, even if the lathe is set on concrete. If at any time the lathe does not bore straight, this is an indication that the lathe is no longer level and the shims should be readjusted.

Testing the Leveling of the Lathe

The accuracy with which the lathe was leveled can be tested by placing a bar of steel 1 in. or more in diameter in the chuck and machining two collars of equal diameter 3 or 4 in. apart. If the collars are not of the same diameter, it is an indication that the lathe is not level. Possibly the level used in setting up the lathe was not sufficiently sensitive. The lathe may be set correctly by adjusting the shims under the front and back legs of the tailstock until the collars on the test piece are turned the same diameter.

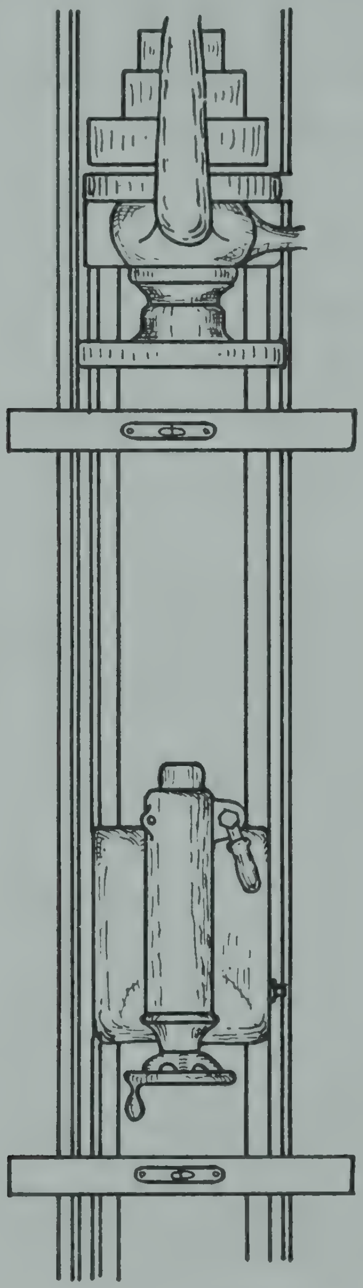


Figure 2. Method of testing leveling of the lathe.

Belts for the Lathe Drive

Flat leather belts are recommended for use on lathe cone pulleys. They have sufficient elasticity to transmit power efficiently and will give excellent service without slipping, provided they are properly adjusted for tension and are kept clean. Leather belts are more efficient and last longer than canvas or rubber belts. If a belt has a tendency to come off the pulley, the alignment of the pulley should be checked. Such a tendency indicates something should be corrected. The belt or the pulley should not be held with a brace.

Notes on Belts and Pulleys. 1. To find the approximate length of a belt, half the sum of the pulley diameters should be multiplied by 31/7 and twice the distance between the pulley centers added.

2. A pulley should be about 10 % wider than the belt.

3. The smooth side of the belt should always run next to the pulley.

4. The belts should be kept clean and dry, and moisture and oil should not be allowed to collect on them.

Belt Tension on Lathe Drive. Maintaining the proper tension on the belt is very important. Belts which are operated loose will creep and slip, reducing the efficiency of the lathe. There is always the danger of damaging fine work because of variation of cutting speeds when operating with a belt which is too loose. Operating a lathe with too much tension on the belt results in loss of power and excessive wear on the bearings, causes the motor to run hot, and stretches the belt, thereby shortening its useful life.

Adjusting Belt Tension on Lathe Drive. To change flat belt tension (Figure 3) on a horizontal motor-driven bench lathe,

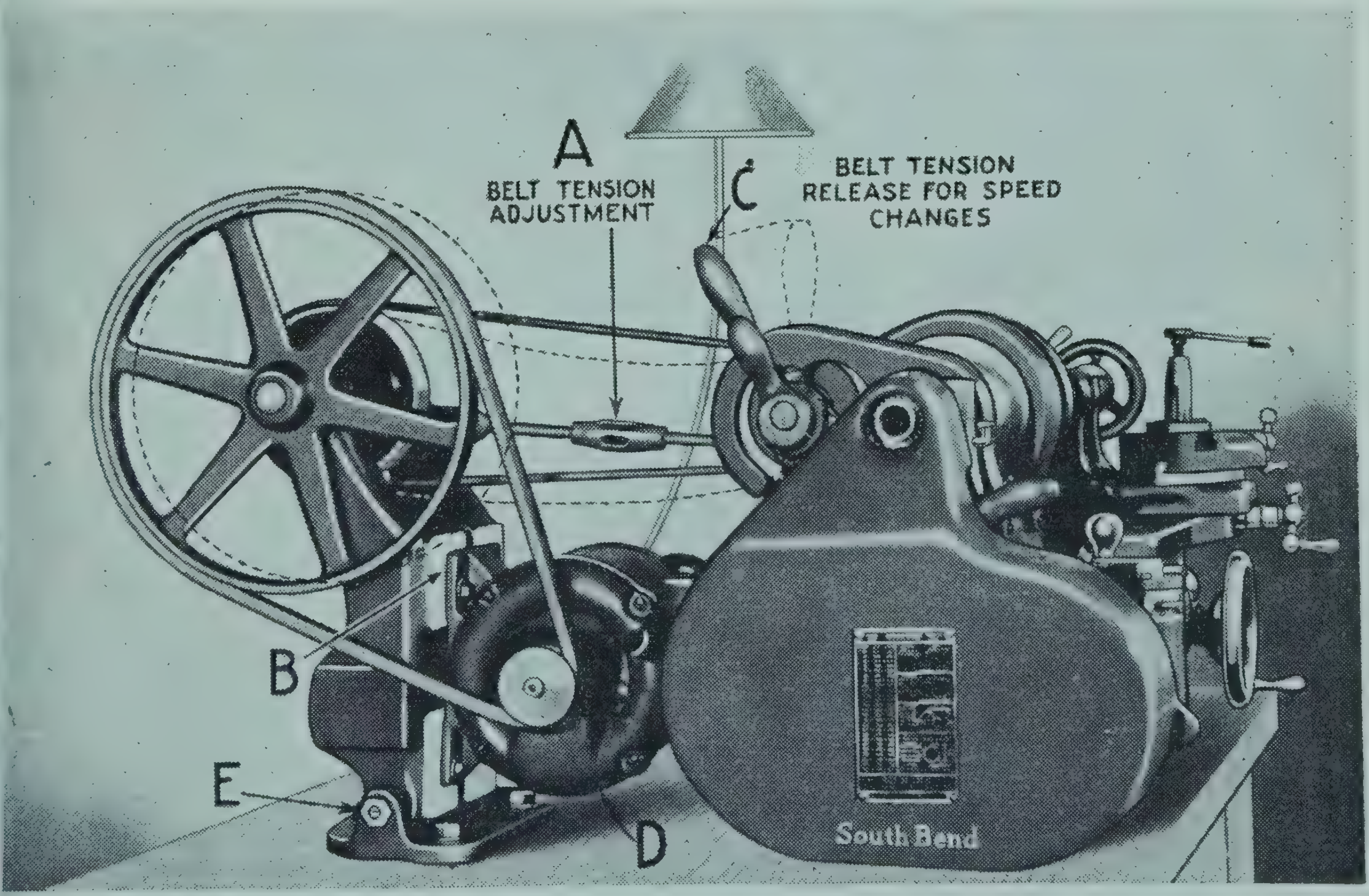


Figure 3. End view of a horizontal moter-driven lathe. (Courtesy of South Bend Lathe Works).

turnbuckle A is adjusted with lever C in the back or down position. In this loosened position the belt should depress about 1 in. If the belt tension is correct and yet it slips under power, it is probably oily and should be cleaned with benzol. If the belt is dry and stiff a little neat's-foot oil will make it soft.

The V belt is adjusted to motor by moving the motor on its mounting. To do this four screws at B (Figure 3) are loosened and the motor moved to give the proper tension. When the tension is correct the belt should depress about 1 in. between pulleys, but it must still be stretched tightly enough to feel alive when tapped lightly.

Oiling the Lathe

Every bearing of a new lathe should be oiled before starting it. Automobile engine oil should never be used to oil a lathe. A high-grade machine oil of the proper viscosity as specified by the manufacture and listed on the lubrication chart attached to most lathes should be used.

Excessive oil should not be used. A few drops in each oil hole is sufficient. If more is applied it will only run out on the lathe. A good procedure is always to oil in the same order so that no oil hole is missed.

Spindle Speeds

The speed with which the spindle rotates is changed by shifting the belt from one step to another on the cone pulley, and by engaging or disengaging the back gears. It is not recommended that back gears be engaged when the drive shaft is operated at high speed.

Principal Operating Parts of the Lathe Carriage and Apron

Figure 4 shows the principal operating parts of a lathe carriage and apron. The carriage is moved along the lathe bed by turning the handwheel, and the tool rest is moved by turning either the compound rest knob or the cross-feed knob. The carriage is locked to the lathe bed by means of the carriage lock screw, which is never tightened, however, except during facing

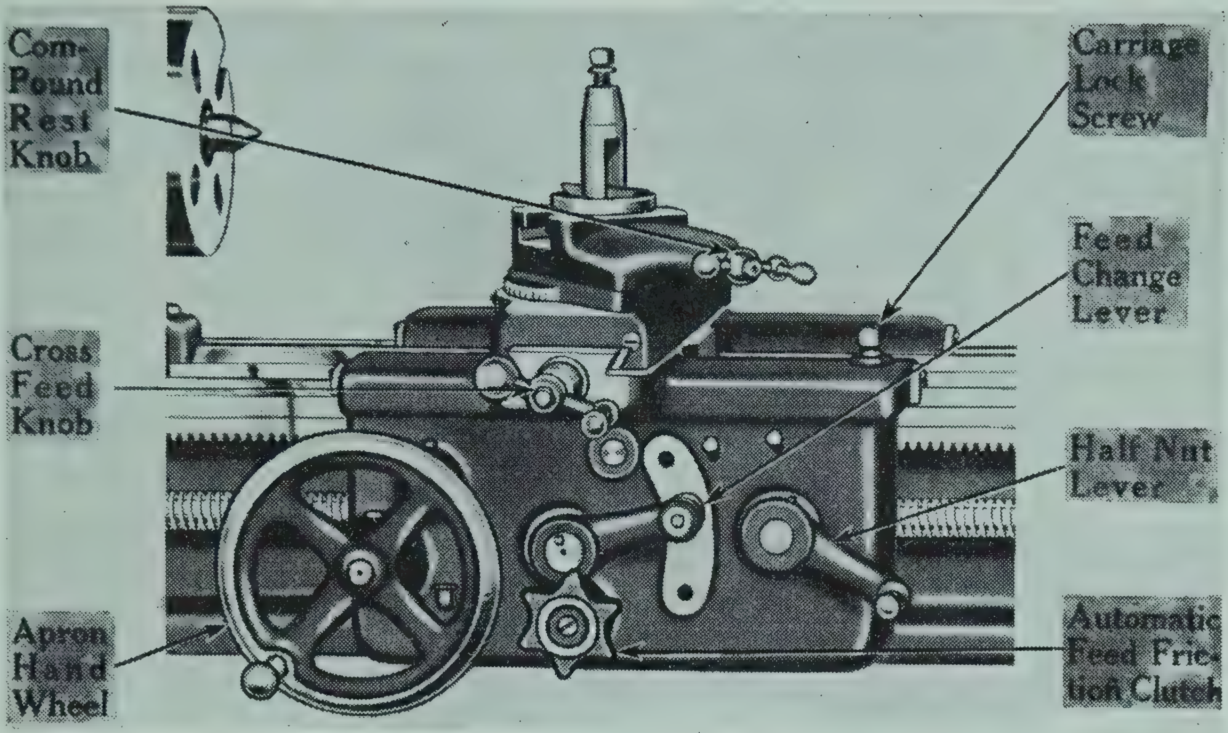

Com-Pound Rest Knob
Cross Feed Knob
Apron Hand Wheel
Carriage Lock Screw
Feed Change Lever
Half Nut Lever
Automatic Feed Friction Clutch

Figure 4. Lathe carriage and apron. (Courtesy of South Bend Lathe Works.)

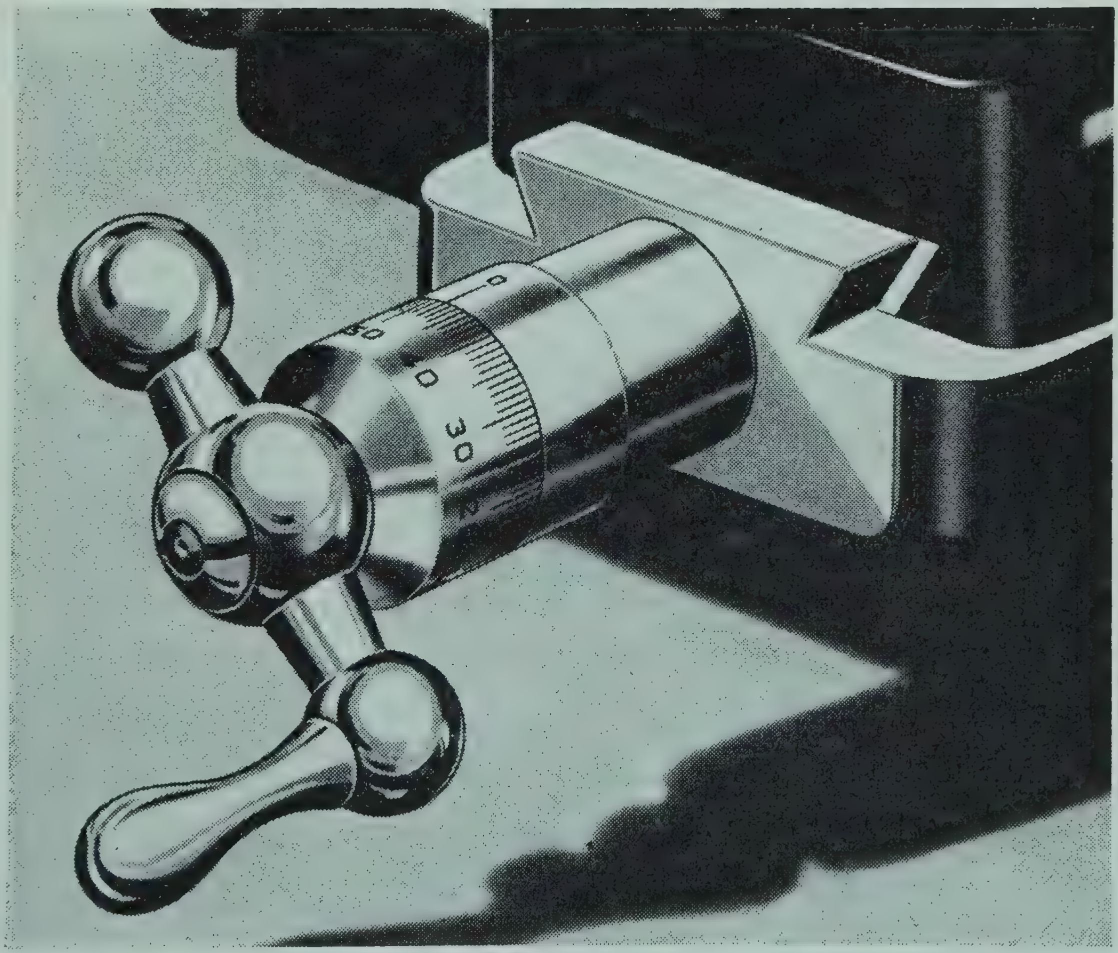


Figure 5. Micrometer collar. (Courtesy of South Bend Lathe Works.)

or cutting-in operations. The automatic feed friction clutch controls the operation of both the power longitudinal feed and the power cross-feed. It is engaged by turning to the left, and disengaged by turning to the right. The feed change lever has three positions: "up" for longitudinal feeds, "down" for cross-feeds, and a center position which is neutral. The half nut lever is used only for thread-cutting operations, and the feed change lever must be in neutral position before the half nut may be engaged.

Micrometer Collars

Each graduation of the micrometer collars on the compound rest knob and the cross-feed knob, as illustrated in Figure 5, represents a movement of one-thousandth of an inch of the compound rest. The graduated collars may be set at zero by loosening the set screws which lock them in position.

FUNDAMENTAL LATHE OPERATIONS

Lathe work may be divided into two general classifications: lathe work between centers and lathe work held in a chuck.

Drilling Center Holes between Centers

Center drilling is the process of drilling and countersinking a hole with a cone-shaped opening to provide a bearing for lathe centers on which the work revolves. Center holes may be located by use of the center gage with hermaphrodite calipers, or with V-blocks and surface gage. After the center holes have been located and the center punched, they are ready to be drilled on the lathe.

Use of a Center Square. The work should be gripped in a bench vise. A line is scribed as shown in Figure 6A. The center square is then turned over and a parallel line drawn as shown in Figure 6B. The center square is then turned one quarter or 90° and two more lines are scribed at right angles to the first pair of lines (Figure 6C). The center punch is then located in the center of the square formed by the four scribed lines.

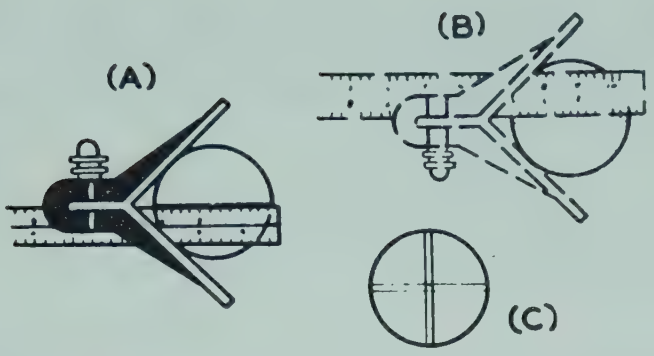


Figure 6. Use of a center square.

Use of a Hermaphrodite Caliper. The work should be gripped in a bench vise. The caliper is set to approximately one-half of the diameter of the work. With the leg of the caliper against the outside of the work an arc is scribed. Three additional arcs are scribed, one from each quarter of the circumference. The center punch is then made in the center of the space which lies between the four arcs (Figure 7).

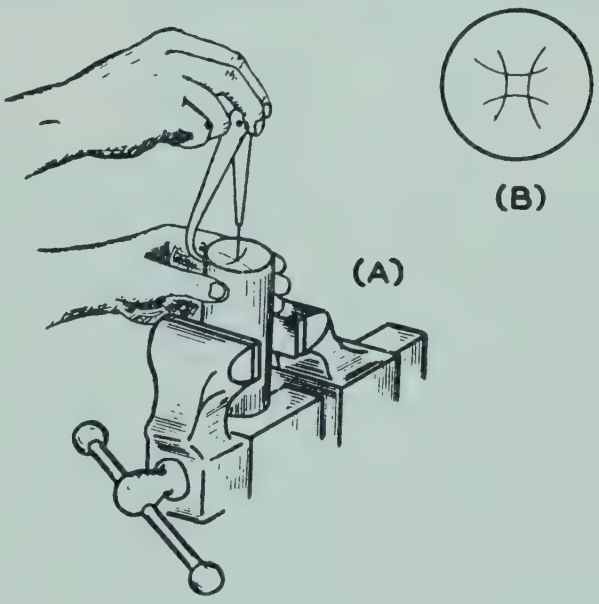


Figure 7. Use of hermaphrodite caliper.

Testing and Drawing Center Points

The lathe is set up with head- and tailstock centers and the work placed between centers as shown in Figure 8A. The tailstock center should be adjusted to support the piece and permit it to turn freely. The work should be revolved with the left hand and the high spots at each end of the work marked with a piece of chalk held in the right hand. If the work does not run true it should be gripped in the vise again and the center punch mark drawn in the direction of the high side. (See Figure 8B.) The work is again tested in the lathe and the operation of drawing the center punch towards the high spot repeated until the work runs reasonably true.

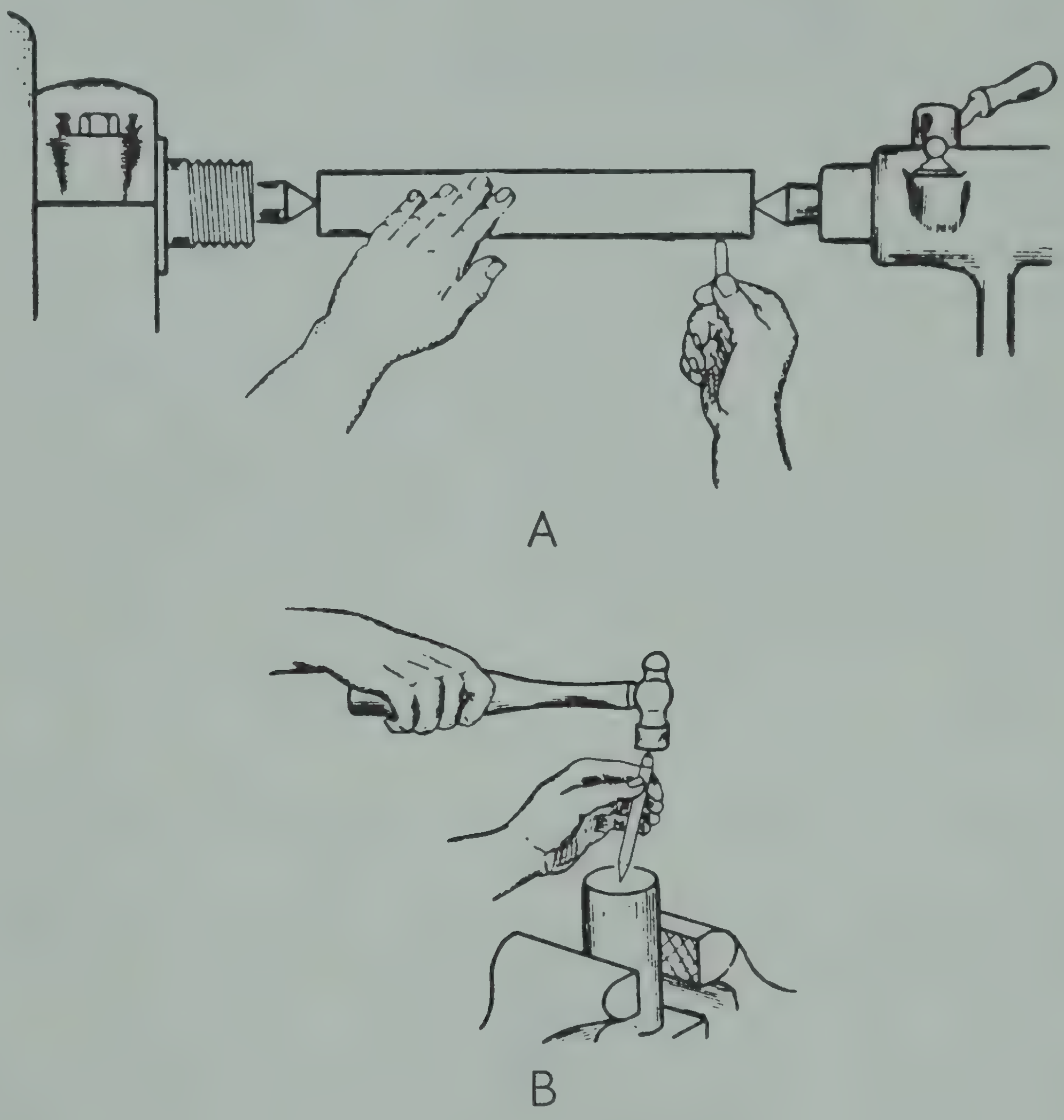


Figure 8. Testing and drawing center points.

Drilling Operations

1. The combination drill and countersink should be gripped in the chuck and tested to make sure it runs true.

2. The tailstock should be checked for the proper alignment and then clamped at the required distance.

3. The work is held in the left hand and the center punch mark placed against the tailstock center. The right hand is used to move the tailstock spindle by turning the handwheel until the drill enters the opposite punch mark.

4. A few drops of oil should be placed on the drill point before starting the lathe. Table I gives the approximate spindle speeds for drilling.

5. The work is held firmly against the tailstock center and slowly fed against the drill until the hole is countersunk to the required depth.

a. Much of the breakage of center drills can be avoided by feeding the work slowly, removing the chips from the drill, and supplying plenty of lubrication.

b. Center holes may be drilled and countersunk by first drilling a pilot hole with a center drill and then countersinking it with a center reamer.

Removing a Broken Center Drill

Center drills which break and remain in the work may sometimes be jarred loose by tapping. In other cases the broken piece may be removed with a punch. When neither of these methods works the end of the piece being drilled should be annealed so as to soften the drill point. After the annealing, the broken piece may be drilled out.

Lathe Work Mounted between Centers

A large percentage of all lathe work is held between centers. Having center holes drilled in the work permits it to be removed from the lathe and replaced later for additional machining operations without disturbing the trueness of the machined parts in relation to the center holes.

Lathe Centers. 1. Lathe centers are known as line centers and dead centers. The line center is mounted in the headstock, supports the left end of the work, and turns with it. The dead center is held in the tailstock, and supports the right end of the work as it rotates.

2. Centers are usually made of a high-grade tool steel and are usually hardened, tempered, and accurately ground, although the headstock center is not always hardened since it acts only as a guide in the rotating and not as a bearing for the work to turn on.

3. The center point is turned and ground to an included angle of 60° to conform to the angle of the center holes drilled in the work. The shank, or opposite end, is tapered to fit the tapered holes in the headstock and tailstock spindles.

Lathe Dogs and Driving Plates. Lathe work which is supported between centers is rotated by a lathe dog which is secured to the work and driven by a driver plate. See Figure 9.

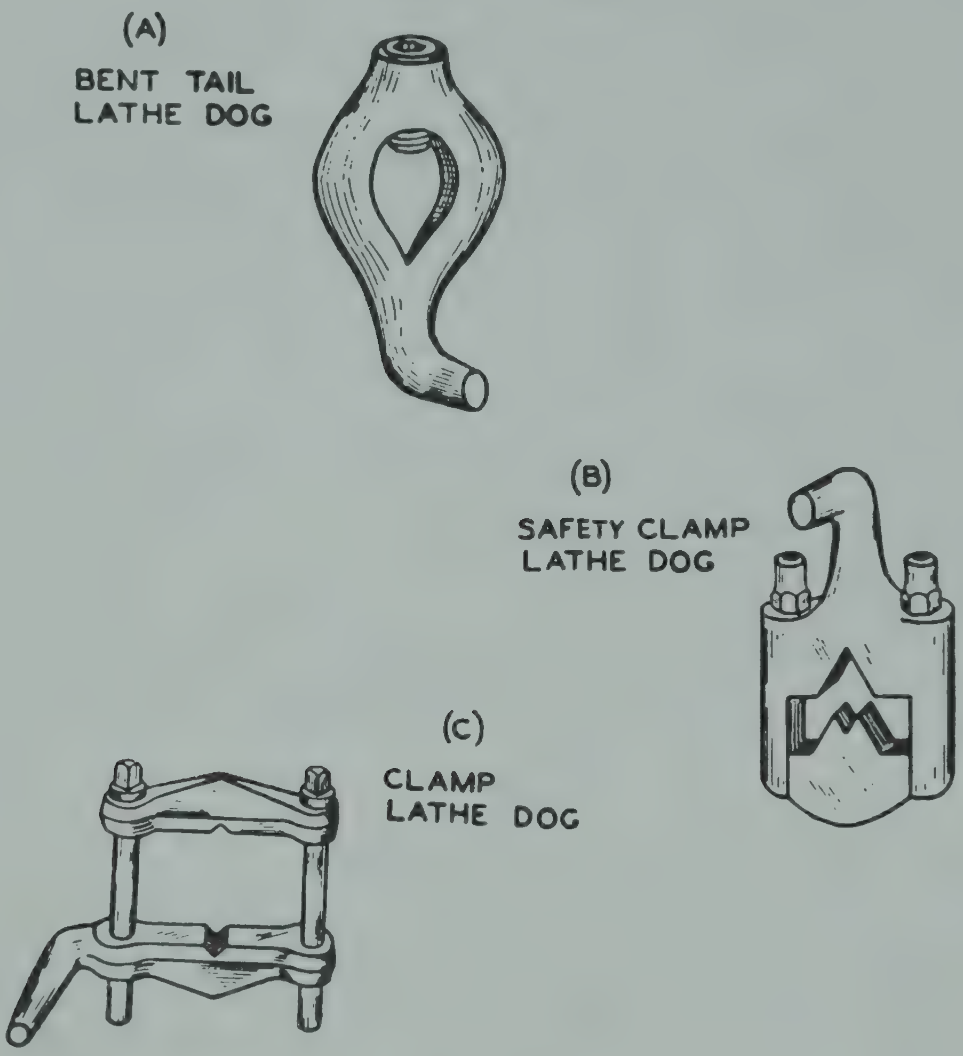

(A)
BENT TAIL
LATHE DOG
(B)
SAFETY CLAMP
LATHE DOG
(C)
CLAMP
LATHE DOG

Figure 9. Common types of lathe dogs.

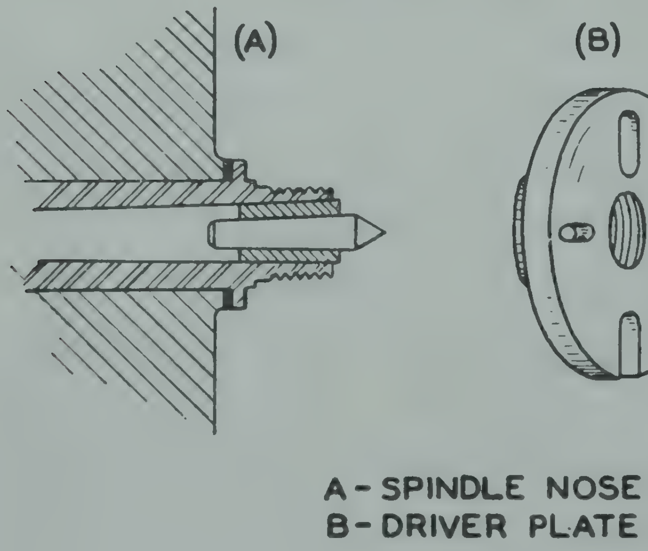

(A)
(B)
A - SPINDLE NOSE
B - DRIVER PLATE

Figure 10. Driving plate.

A lathe dog has two principal features, an opening which receives the work and a bent tail which fits in the slot of the driving plate. The bent tail of the dog transmits the driving force from the driver plate (Figure 10) to the work.

Mounting Work between Centers. Work that is to be turned accurately must be mounted correctly in the lathe in order to provide a good bearing and support for the center holes of the work. There are several important factors to be considered in properly mounting the work.

1. Alignment of the lathe centers. (*a*) The approximate alignment of the lathe centers may be ascertained by sliding the tailstock forward until the two centers nearly meet. (*b*) Any error in alignment may be corrected by adjusting the set-over screws as shown in Figure 11.

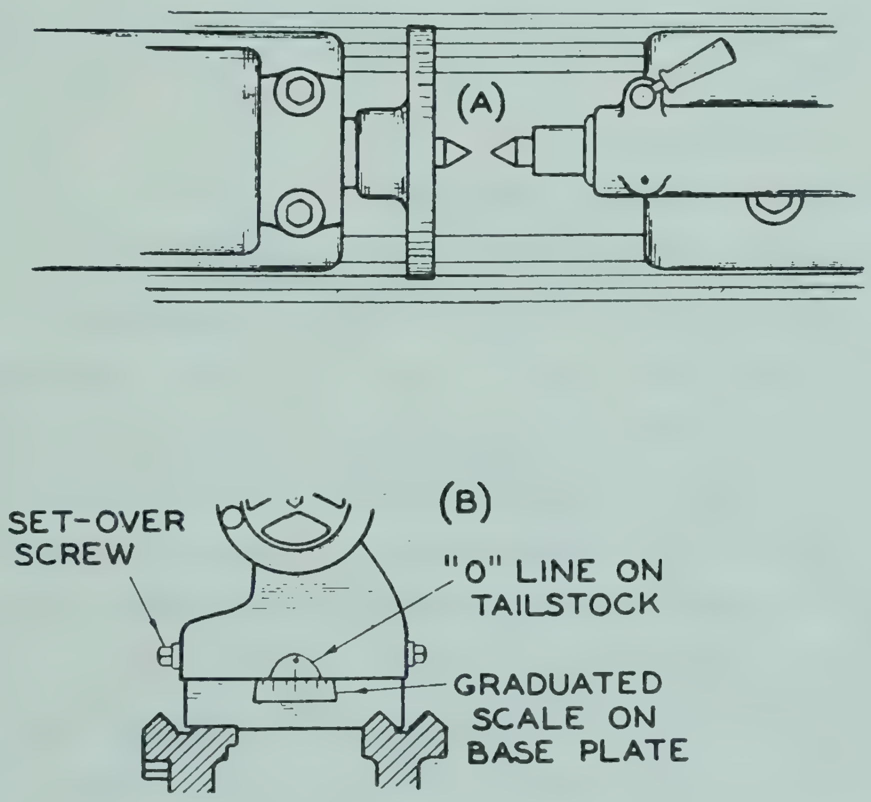


Figure 11. Adjusting centers.

2. The trueness of the center holes.
3. The lubrication of the tailstock.
4. The proper adjustment of the lathe dog.

Note: The finished surface of work should be protected with a piece of soft brass or copper so that the set screws will not mar the work when they are tightened.

Procedure for Mounting Work between Centers

The work to be mounted between centers should be held in the left hand and the countersunk hole placed over the headstock center so that the tail of the dog is freely engaged in a slot in the driver plate. In case the dog does not enter the slot freely, the set screw should be loosened and the dog adjusted so that the tail does not bind against the sides of the slot. Care should also be taken in mounting the work to be sure that the tail of the dog does not rest on the bottom of the cored slot in the driver plate.

The tailstock center is moved toward the work by turning the tailstock handwheel with the right hand. When the work is supported by the centers, adjustment should be made so that no play can be felt between the work and the centers. At the same time, the work should be free enough so that the weight of the dog will cause it to move when the dog is moved to one side of the slot.

Lathe Work Mounted in Chucks

Although many operations commonly done in a lathe are performed with the work held between centers, many of these operations can be performed better and more quickly when the work is held in a chuck. Round pieces are usually held in a universal chuck because the jaws are self-centering and, when tightened, hold the work concentric. The accuracy with which the work is held concentric depends on the condition of the jaws and the surface to be chucked.

Description of Chuck. A chuck is a device, which, when fixed to the spindle of a machine, is used to hold either a tool or the material to be worked on. The jaws of the chuck do the gripping, and they are adjustable so that work of different sizes may be held. The jaws are operated either by screws or by a gear-driven scroll.

The *universal chuck* is provided with three accurate self-centering jaws, which are controlled by a bevel gear-driven scroll. All three jaws are moved by turning any one of the three adjusting sockets. The jaws of a universal chuck are not reversible as are the jaws of an independent chuck. For this reason two sets of jaws are usually furnished with each chuck to accommodate either small- or large-diameter work and work which requires inside chucking.

2. The *independent four-jaw chuck.* As the name implies, each jaw of this chuck can be adjusted independently of the others. The jaws are also reversible and stepped to accomodate work of large diameter. The independent four-jaw chuck is a very adaptable type of chuck because it can be used for holding any shaped piece, either concentric or off-center, as the job requires.

3. The *combination chuck* embodies the mechanical features of both the universal chuck and the independent jaw chuck. It is so constructed that adjustment of the jaws may be made either independently by turning the adjusting screws for each jaw, or universally by turning the adjusting socket which operates the bevel gear-driven scroll.

Mounting a Chuck on a Lathe. 1. Before any attempt is made to mount a chuck on a lathe, the threaded hole of the chuck plate should be throughly cleaned to remove any metal chips which may have become lodged in the threads.

2. The threads on the spindle nose should also be brushed free of chips and wiped.

3. The chuck should be supported in the crook of the right arm and lined up with the lathe spindle while the left hand is used to turn the lathe spindle and engage the threads. The chuck is then screwed on to the spindle nose by hand until it shoulders. A chuck should never be forced too tightly against the shoulder, and under no circumstances should the chuck be mounted by using power.

Removing the Chuck from the Lathe. The jaws should be extended past the outside diameter of the chuck and a block of wood placed on the back ways of the lathe directly under the extended jaw as shown in Figure 12. With the back gears of the lathe engaged to give added power, the spindle should be turned by hand in the reverse direction, or clockwise, until the extended jaw strikes the wooden block sharply and jars the chuck loose. Under no circumstances should a chuck be removed by turning on the power after it has once been started. The chuck should be well supported to prevent damage to the threads on the spindle nose or damage to the ways of the lathe from dropping the chuck on them.

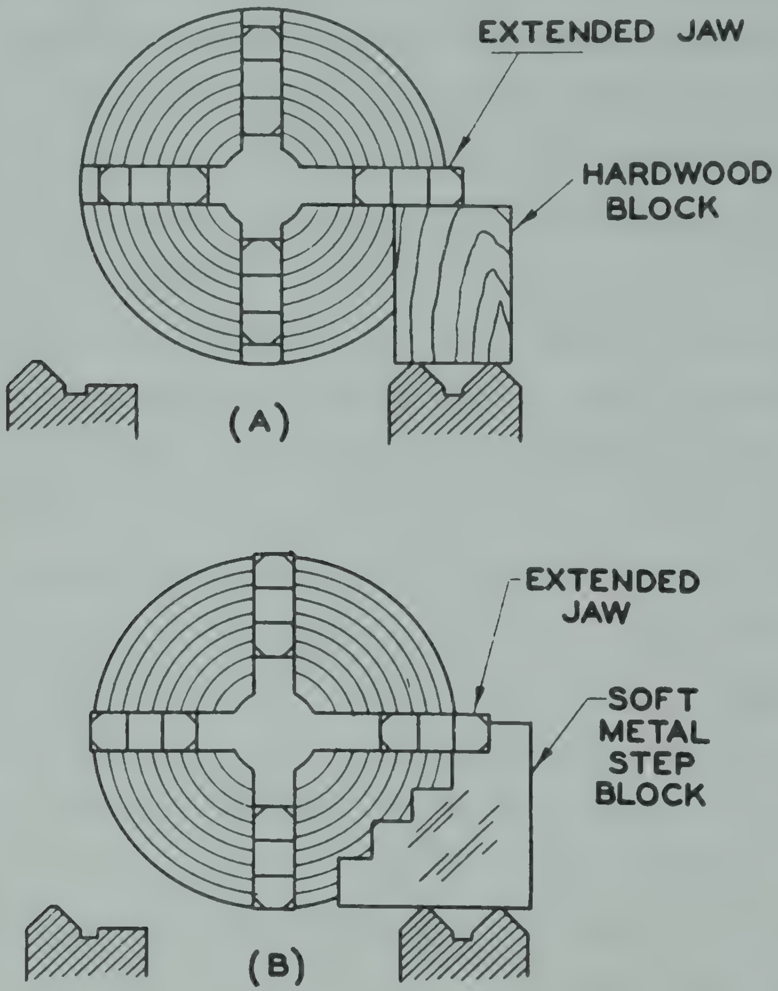


Figure 12. Removing the chuck from a lathe.

Changing Jaws in a Universal Chuck. Each jaw of the universal scroll chuck is marked with a number which corresponds to the same number stamped near the slot on the body of the chuck. If the jaws of the chuck are changed or removed for any reason, they should be assembled in the slot bearing the number of the jaw. Only one jaw can be started in the scroll at one time; however, the jaws should be assembled in the chuck in their proper order so that they will be concentric. Jaw 1 is engaged first; the scroll is then turned, engaging jaw 2, and finally jaw 3.

Trueing Work in a Four-Jaw Independent Chuck. The jaws of the chuck are adjusted to the approximate size of the job to be held; the position of the jaws is judged by the ring marks on the face of the chuck (see Figure 13). The work is gripped by tightening the pair of jaws which are opposite each other (1 and 3), then tightening the other pair (2 and 4). Work of large diameter may be seated against the face of the chuck. The work should be chucked just tightly enough to hold it in the chuck, since further adjustment probably will be necessary. When a round piece is being chucked, the jaws should be inspected to see that they are all located the same distance from the grooves marked in the face of the chuck. The spindle is then turned slowly to determine if the work runs approximately true. High spots may be marked with a piece of chalk as shown in Figure 14. The chalk is held steady by resting the arm on the lathe or tool post rest to overcome the tendency of the chalk to move in and out with the eccentricity of the work.

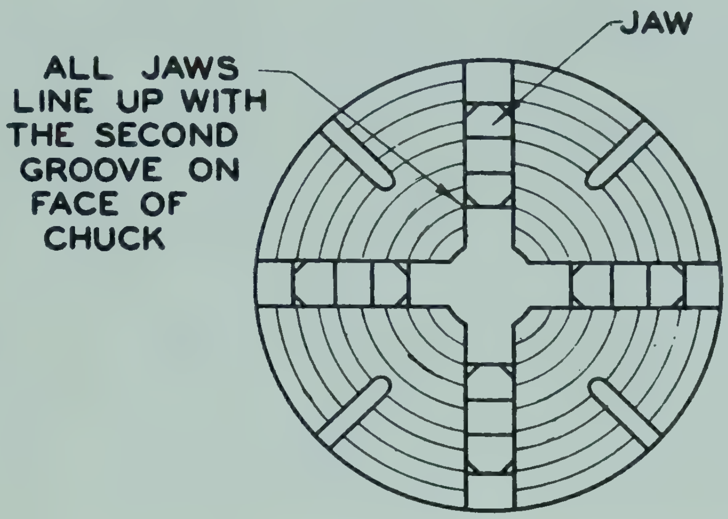


Figure 13. Trueing work in a four-jawed chuck.

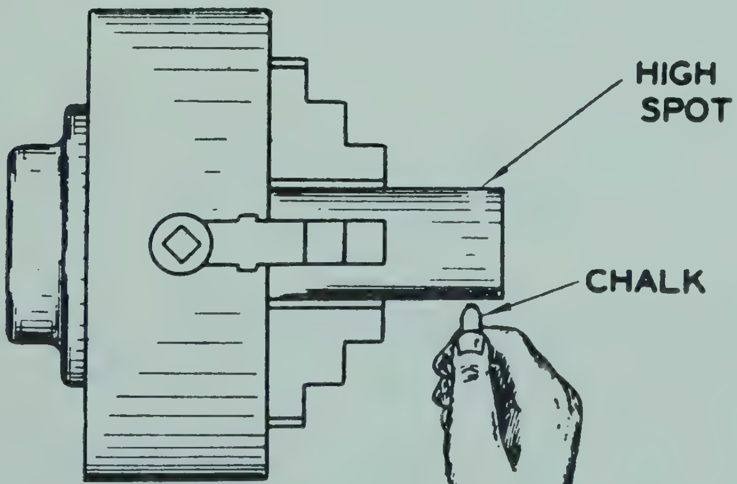


Figure 14. Marking high spots with chalk.

TABLE I. Cutting Speeds for Turning, Drilling, and Tapping.
(With high-speed steel cutting tools)

Material	Turning speeds		Drilling speeds		Tapping speeds	
	Ft./min.	Lubricant	Ft./min.	Lubricant	Ft./min.	Lubricant
Aluminum	300–400	Comp. or kerosene	200–300	Comp. or kerosene	90–110	Kerosene lard oil
Brass, leaded	300–700	Dry or comp.	200–500	Comp.	150–250	Comp. or base oil
Brass, red and yellow	160–300	Comp.	75–250	Comp.	60–150	Comp. or base oil
Bronze, leaded	300–700	Comp.	200–500	Comp.	150–250	Comp. or base oil
Bronze, phosphor	75–150	Comp.	50–125	Comp.	30–60	Comp. o
Cast iron	50–110	Dry	100–165	Dry	79–90	Dry or c
Cast steel	45–90	Comp.	34–45	Comp.	20–35	Sul. bas
Copper, leaded	300–700	Comp.	200–500	Comp.	150–250	Lt. base
Copper, electro	75–150	Comp.	50–125	Comp.	30–60	Lt. base
Chrome steel	65–115	Comp.	50–65	Comp.	20–35	Sul. bas
Duralumin	275–400	Compound	250–375	Comp.	90–110	Comp. or and lar
Monel metal	100–125	Comp. or sul. base	45–55	Sul. base	20–30	Sul. bas kero. & oil
Nickel silver 18 %	75–150	Comp.	50–125	Comp.	30–60	Sul. bas kero. & oil
Nickel silver leaded	150–300	Comp.	75–250	Comp.	60–150	Sul. bas kero. & oil
Nickel steel	85–110	Comp. or sul. base	40–65	Sul. base oil	25–40	Sul. bas
Stainless steel	100–150	Sul. base	30–45	Sul. base	15–30	Sul. bas

Comp. = Compound; Sul. = Sulfur; Kero. = Kerosene; Lt. = Light.

To correct a high spot, the two jaws opposite the chalk mark should be released and the other two jaws tightened. One set of jaws is adjusted at a time so that the work will not fall out of the chuck.

Testing and adjusting is continued until the work runs true. In trueing, it will be noticed that the work is sometimes "skewed" in addition to running out eccentrically, that is, the work may be concentric near the chuck while the end runs out. This may be corrected by tapping the high side lightly with a lead mallet.

Testing for Trueness with an Indicator. When work must be accurately trued, an indicator held in a tool post may be used to indicate any eccentricity (Figure 15). The contact point of the indicator is carefully brought into contact with the work and the chuck is revolved slowly. The amount that it runs out of true is indicated by the movement of the pointer. The high spot is marked and the jaws adjusted until the work runs true.

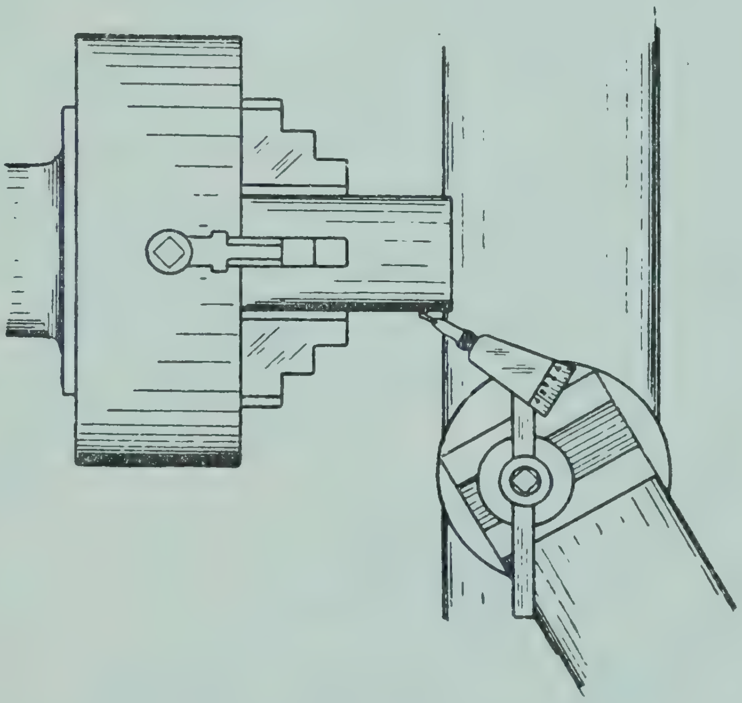


Figure 15. Testing the trueness with an indicator.

Drilling Center Holes in Work Held in a Chuck. Center holes are drilled in the end of work to provide either a bearing surface for lathe centers or a pilot hole for subsequent drilling operations. (See Table I for spindle speeds for drilling.)

1. The drilling of center holes for chuck work differs from drilling holes between centers in that the work revolves while the

drill or countersink is held in a drill chuck and fed into the work.

2. While the combination drill and countersink or center drill is most frequently used for centering, there are times when a starting hole can be spotted with either a flat drill held in a drill chuck or a spotting tool mounted in the lathe tool post.

3. Work to be centered should not extend beyond the chuck any farther than is necessary. Excessive overhang causes the lathe to vibrate and results in drill breakage.

4. The end of work to be centered should be faced square.

5. After the drill has been brought up to the face of the work, it should be noted whether the point centers in the initial centering hole. If it does not, it is out of alignment and the tailstock should be adjusted to permit the drill to center properly, thus preventing drill breakage.

6. The drill should be properly lubricated with cutting oil.

Spotting a Center. In place of the combination drill and countersink, a spotting tool may be used. A more modern method is to grind a special tool bit for centering (see Figure 16). The side and front of such a tool is ground with sufficient clearance to permit the tool to punch a solid piece. This centering tool may also be used to true a center spot which has run out. Regardless of how the centering is done, care must be taken to make sure the "spot" center is not larger than the hole to be drilled.

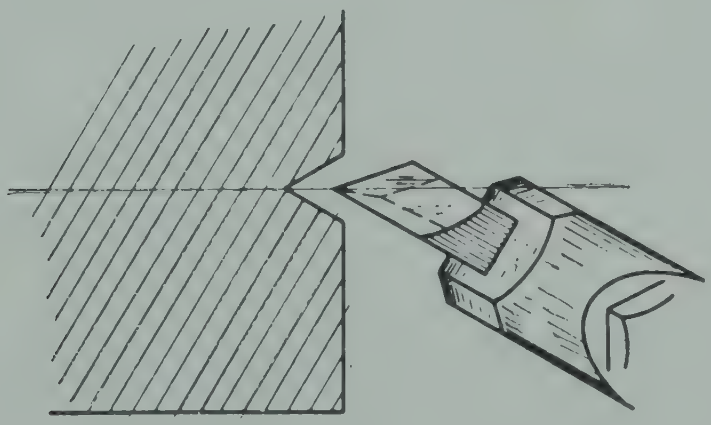


Figure 16. Centering with a specially ground tool bit.

Notes on Operations Requiring Drilling. 1. In chuck work a hole is frequently drilled to precede other machining operations such as boring, reaming, and tapping. Although a drill press is ordinarily used for drilling holes, certain types of jobs can be done more easily on a lathe.

2. Work is drilled on a lathe in much the same way as in a drill press except that in chuck work the work revolves instead of the drill.

3. In drilling deep holes, the drill should be backed out of the work occasionally to remove chips from the drill in order to prevent clogging and drill breakage.

Boring

Boring should not be confused with drilling. Drilling usually refers to cutting a hole in a solid piece of material, whereas boring refers to enlarging a drilled hole. Boring on a lathe could be

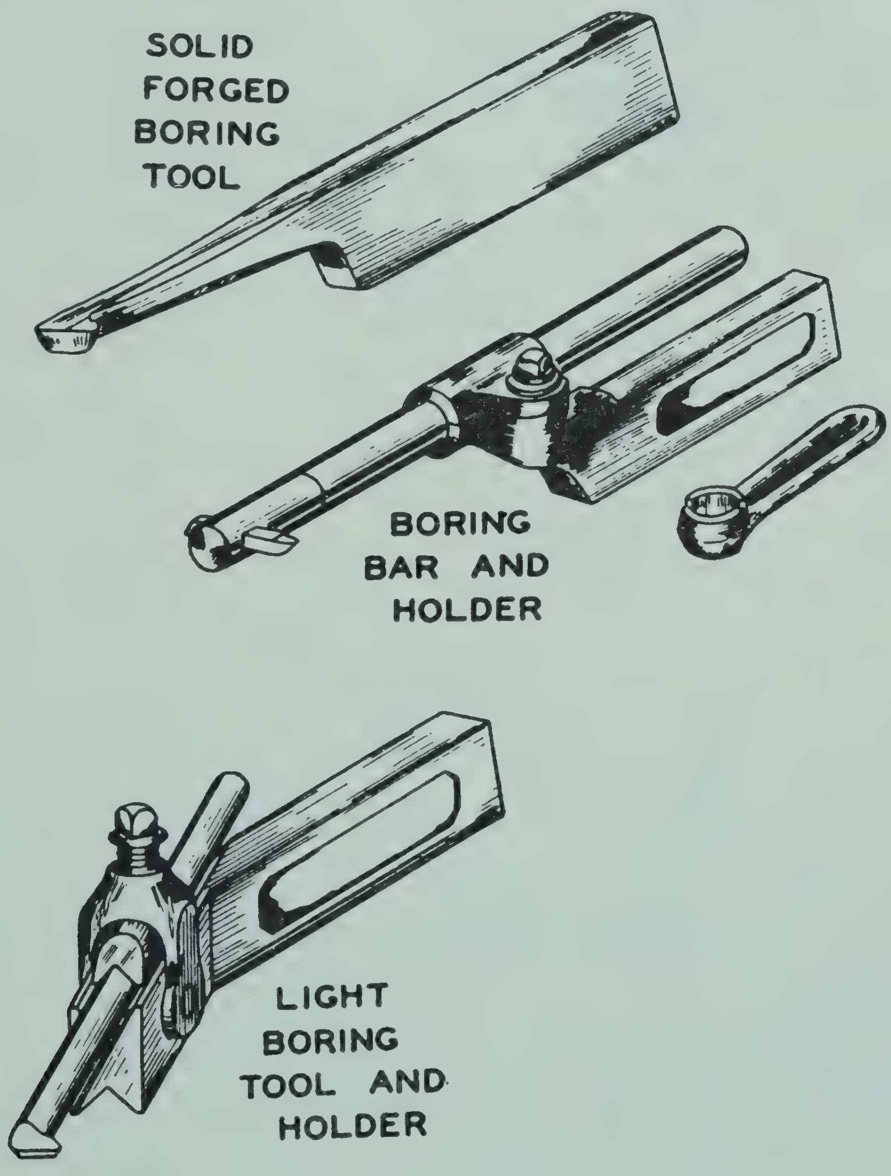
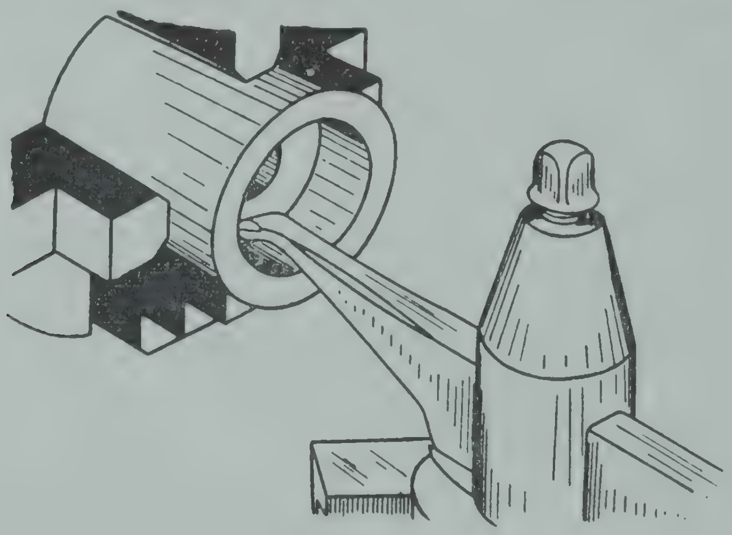

SOLID FORGED BORING TOOL
BORING BAR AND HOLDER
LIGHT BORING TOOL AND HOLDER

Figure 17. Boring tools.

referred to as an internal turning operation as compared to external turning on an outside diameter. Boring tools are available in a variety of different types. Some of these are made (see Figure 17) from a solid bar. A more common practice is the use of a boring bar in which high-speed steel cutter bits are inserted. The boring bar has a decided advantage over the solid forged tool since it is held in a holder and the amount of overhang can be adjusted to suit the length of the hole being bored. This provides the greatest amount of rigidity and eliminates vibration and chattering of the tool. Another advantage of the boring bar is that the inserted tool may be removed, resharpened, and replaced without disturbing the position of the holder.



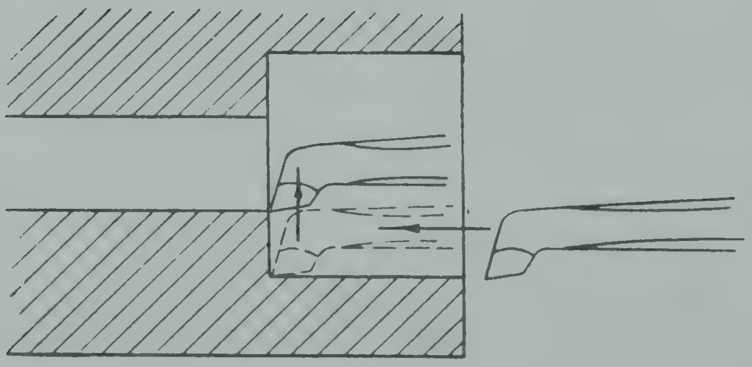


Figure 18. Counter boring.

Boring Operation. 1. The boring operation is usually preceded by drilling unless the work is already provided with a hole.

2. The largest size boring tool which will fit in the hole should be used to insure rigidity.

3. Boring is the more dependable method of producing holes which are concentric. Boring should also be used to true up holes which are not concentric.

4. A good grade of cutting oil should be used in boring to obtain a smooth finish cut.

5. The diameter of the hole should be measured carefully to see that it is not "bell mouthed." If so, additional cuts of the same setting of the tool should be made to produce a parallel bore.

6. If the boring tool chatters, the spindle speed should be reduced. The gibs on the cross slide and compound rest should be inspected and adjusted to eliminate all play.

Counterboring. Counterboring is another form of boring on a lathe and refers to enlarging the front of a bored hole to a definite size for a given depth. See Figure 18.

Tapping on a Lathe

The tapping of chuck work on a lathe is similar to tapping on a drill press. The shank end of the tap is guided by the tailstock center, which aligns the tap true with the drilled or bored hole. This assures a thread whose sides are parallel to the hole when the tap is turned into the work.

Procedure. The spindle should be locked to prevent its turning while the tap is turned into the work and at the same time the tailstock handwheel is turned so that the center follows the

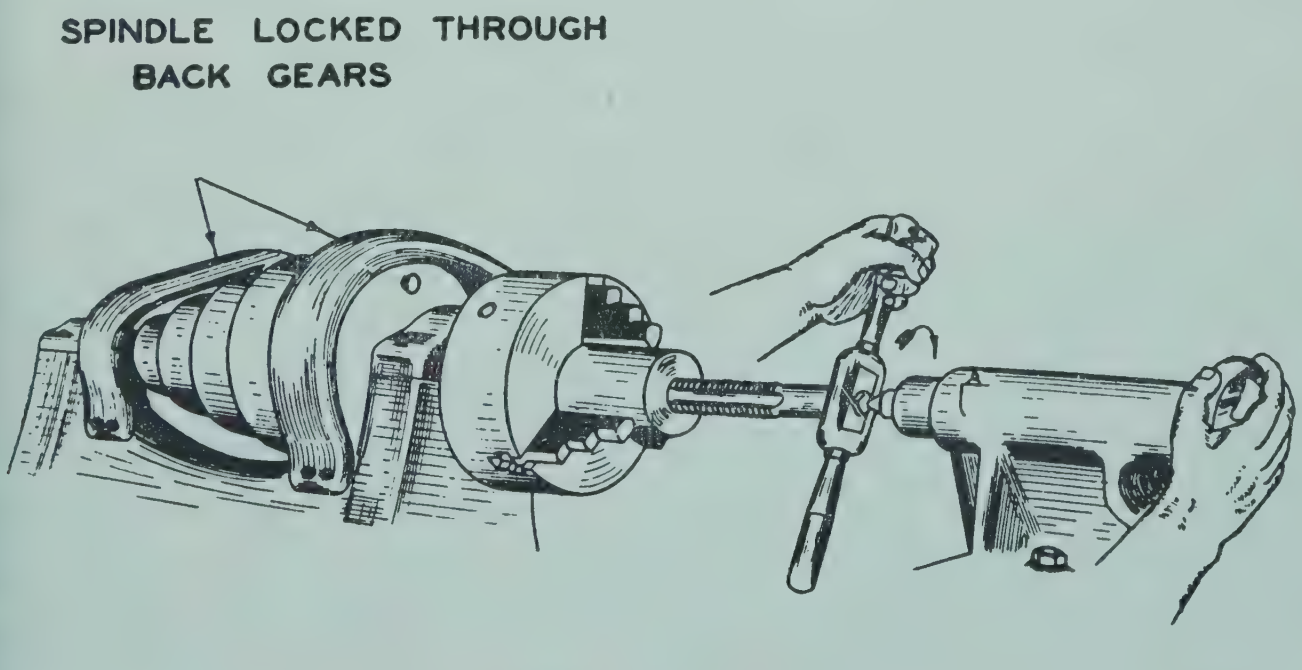


Figure 19. Tapping on a lathe.

tap as it advances (see Figure 19). Enough pressure should be exerted at the start to make the tap catch the thread and prevent it from merely reaming out the metal at the front of the hole. Since a tap that has started will feed itself into the work when turned, the tailstock center is used only as a guide to keep the tap in alignment. After each turn of the tap wrench, the pressure of the tailstock center should be released and the tap backed up slightly to break the chip.

Blind holes, or holes that do not go all the way through the work, should be tapped with a bottoming tap in order to cut threads the full length of the hole. Care should be exercised in tapping blind holes to avoid forcing the tap into the bottom of the hole and causing it to break.

Cutting Off Stock on a Lathe

Cutting off or parting on a lathe is the operation of cutting a groove around a piece of work in order to sever it from the piece held in the lathe, and most cutoff operations are performed on work held in a chuck or collet. The cutoff tool is held in the lathe tool post and is turned into the work until the groove made by the tool completely separates the work. The cutoff tool is always set at right angles to the center line of the work and is fed to the work by the movement of the cross slide.

Some of the applications for this operation are cutting off parts which have already been turned on the lathe and the cutting off of tubing or bar stock to the required length.

Tools. The cutting-off tools are shown in Figure 20. These are ground so that the widest part of the tool is the cutting edge. The sides of the blade are beveled to provide side clearance, with the blade wider at the top than the bottom. A difference of about 2° is usually sufficient to allow the tool to clear the sides of the groove being cut, and the same clearance (about 2°) should be allowed for back clearance. The front clearance of the tool should be about 15° for soft materials such as brass and 10° for harder materials like cast iron, cold drawn steel, and tool steel. The top of the tool is usually ground flat for soft materials because of the tendency of the tool to dig into the work. For harder materials a back rake of 5° tends to curl the chip and help the tool cut freely.

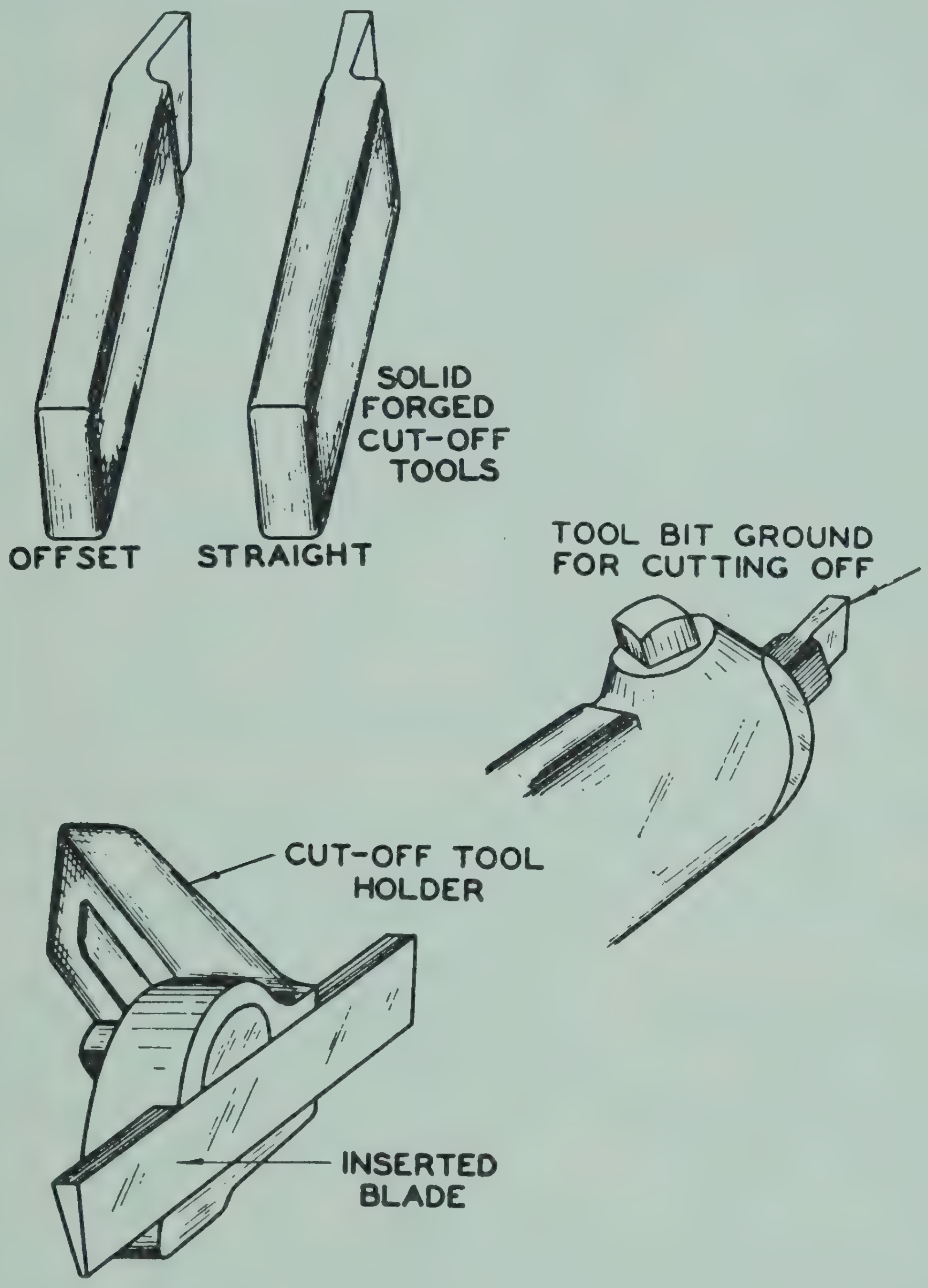

SOLID FORGED CUT-OFF TOOLS
OFFSET
STRAIGHT
TOOL BIT GROUND FOR CUTTING OFF
CUT-OFF TOOL HOLDER
INSERTED BLADE

Figure 20. Cutting-off tools.

Speeds. The cutting speed for cutoff tools is generally governed by the hardness of the metal being turned, the kind of steel from which the tool itself is made, the width of the cutting tool edge, the cutting compound, and even the condition of the lathe. Generally the spindle speeds for cutting off are approximately one-half to two-thirds the speed used for turning the same materials. See Table IIA and Table IIB for cutting speeds and tool angles.

TABLE IIA. Cutting Speeds and Tool Angles for Nonferrous Materials

Material	Cutting speed, f.p.m.	Front clearance degrees	Side clearance degrees	Back rake degrees	Side rake degrees
Brass, leaded	300–700	6	7	0	0
Bronze, free cutting	300–700	6	6	0	1½
Bronze, readily machinable	150–300	10	8	7	7
Bronze, tough	75–150	12	15	15	25
Copper, pure	75–150	7	7	10	25
Die castings	225–350	7	7	8	10
Magnesium alloy	275–400	8	8	6	4
Monel metal	50–170	6	6	7	12
Plastics, cast resin	200–600	10	12	30	25
Plastics, cold set molded	200–600	10	10	0	0
Plastics, hot set molded	200–600	6	6	10	30
Plastics, laminated	200–600	8	8	30	30
Stainless steel	50–150	8	8	9	0
Wood	400–800	20	20	30	30

TABLE IIB. Cutting Speeds in r.p.m. for Turning Metals with High-Speed Steel Tools

Material	Turning and boring		Cutting screw threads
	Rough cut 0.10 to 0.20 in.	Finish cut 0.020 to 0.010 in.	
Machine steel	90	100	35
Tool steel annealed	50	75	20
Cast iron	60	80	25
Brass	160	220	60
Aluminum	200	300	50
Bronze	90	100	25

1. The cutting speeds in Table IIB are decreased approximately one-half when tools of carbon tool steel are used.

2. When tungsten-carbide tipped cutting tools are used, the cutting speeds in Table IIB may be increased from 100% to 800%.

3. When a cutting lubricant is using the cutting speeds in Table IIB may be increased by 25% to 50%.

Note: The cutting speed in feet per minute may be found by multiplying the diameter of the work in inches by 3.14, and by the number of revolutions per minute (r.p.m.) and then dividing this product by 12. For example,

$$C = \frac{3.14 \times D \times \text{r.p.m.}}{12}.$$

TABLE III. Difficulties Encountered in Cutting-off Stock Showing Causes and Remedies

Difficulty encountered	Cause	Remedy
Chatter	Speed too great	Decrease cutting speed
	Tool extended too far	Clamp tool "short"
	Play in cross slide and compound rest	Adjust gibs on cross slide and compound rest
	Backlash in compound rest feed screw	Back compound rest away from work by turning feed screw to the left
	Play in lathe spindle	Adjust end play in spindle bearings
Dulling of tool	Cutting speed too high	Decrease cutting speed
	Too much front clearance	Decrease front clearance angle
	Lack of cutting lubricant	Apply lard oil or cutting compound for particular metal being turned
	Overheating and drawing temper of tool due to insufficient clearance	Grind front, side, or back clearance
	Overheating due to excessive cutting speed	Decrease spindle speed
Tool digging into work and work "climbing" on tool	Tool set too low	Set tool at height of lathe center
	Play in compound and cross slide	Adjust gibs on cross slide and compound rest
	Backlash in compound rest feed screw	Back compound rest away from work by turning feed screw to the left
	Too much front clearance	Decrease front clearance angle
	Too much top rake	Decrease top rake
Tool riding on work	Tool too high	Set tool at height of lathe center
	Insufficient front clearance	Increase amount of front clearance angle

TABLE IV. Common Errors in Center Drilling

Error	How to avoid and correct the error
No clearance for point of center	Drill pilot hole
Center hole incomplete	Countersink pilot hole at 60°
Insufficient bearing surface for lathe center	Drill center hole with combination drill and countersink
No bearing surface for center	Countersink mouth of hole at 60°
Insufficient bearing surface for lathe centers	Countersink deeper
Hole drilled too deep with combination drill and countersink	Face end if job will allow
Poor bearing surface	Ream mouth with center reamer
Poor bearing surface Wrong angle	Countersink hole with 60° center reamer
Center hole drilled at angle to the axis of work	Align work squarely when drilling center hole Face end and recenter

Procedure. The cutoff blade should be mounted in the tool post with the cutting edge at the height of the lathe center, and the tool square with the work so that the sides of the blade will not rub on the edge of the groove. See Figure 21. The cutoff tool should be clamped short in the tool post in order to eliminate as much vibration as possible to prevent the tool from chattering and breaking. This is done by adjusting the blade so that it extends only slightly more than half the diameter of the work. *Note:* The carriage should be locked to prevent it from moving during the cutoff operation.

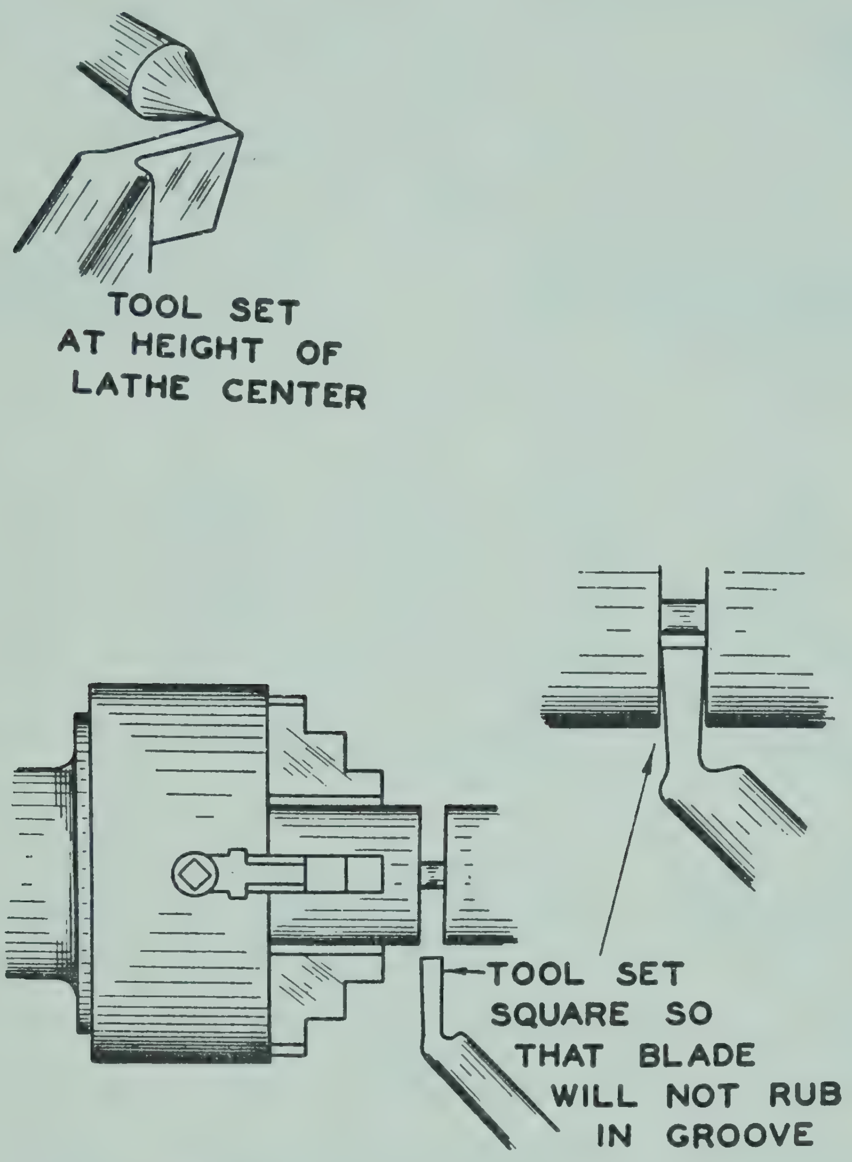

TOOL SET
AT HEIGHT OF
LATHE CENTER
TOOL SET
SQUARE SO
THAT BLADE
WILL NOT RUB
IN GROOVE

Figure 21. Setting up cutting-off tools.

On large-diameter work the lathe may be stopped near the end of the cut, leaving a small amount of stock at the center. The amount should be small enough that the piece may be easily broken off after the cutoff tool has been removed from the cut. See Table III for difficulties encountered in cutting off and remedies for them.

Table IV illustrates some of the difficulties that may be encountered in center drilling, and suggests steps to correct these difficulties.

Lathe Tool Bits

In lathe work the most commonly used tools are the right-

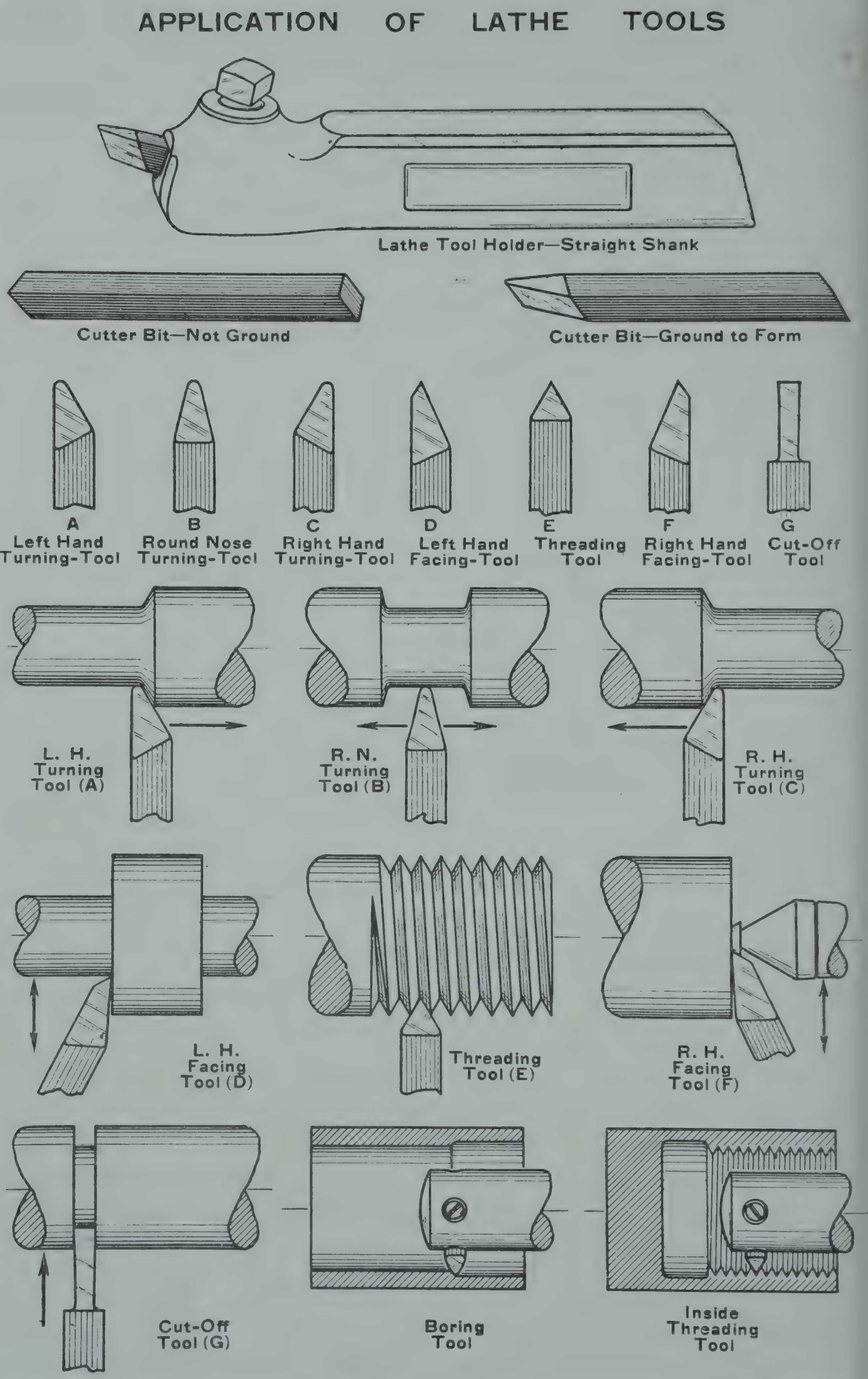

APPLICATION OF LATHE TOOLS
Lathe Tool Holder—Straight Shank
Cutter Bit—Not Ground
Cutter Bit—Ground to Form
A Left Hand Turning-Tool
B Round Nose Turning-Tool
C Right Hand Turning-Tool
D Left Hand Facing-Tool
E Threading Tool
F Right Hand Facing-Tool
G Cut-Off Tool
L. H. Turning Tool (A)
R. N. Turning Tool (B)
R. H. Turning Tool (C)
L. H. Facing Tool (D)
Threading Tool (E)
R. H. Facing Tool (F)
Cut-Off Tool (G)
Boring Tool
Inside Threading Tool

Figure 22. Description of lathe tool bits. (Courtesy of South Bend Lathe Works).

and left-hand turning tools, the right- and left-hand facing tools, the round-nose turning tool, the threading tool, and the boring tool. Figure 22 shows these tools and the operation for which they are most fitted.

High-Speed Tool Bits. These bits are standard lengths and need only to be ground into the desired shape. The 30° angle at the ends of the tool bit serves as a guide for the proper amount of front clearance to the tool when it is placed in the tool holder. The tool fits the holder at an angle of about 20°, leaving a front clearance of about 10°, which is satisfactory for most purposes.

The Rake. The rake of the tool refers to the slope of the tool. This is ground on the top side and slopes away from the cutting edge. The angle of the slopes from the cutting edge toward the side of the tool is called the side rake. The angle that slopes from the front of the tool towards the back is called the back rake.

The combination back and side rake forms a double angle which gives a shearing action to the cutting edge, thus permitting the material to be machined more freely and causing the chip to curl away from the edge which does the cutting. Different metals require more or less rake; in general, mild steels such as cold rolled steel require more rake than hard steels or cast iron.

Clearance. The clearance of the cutting tool is determined by the angle necessary to clear the work as the tool is fed into it to make the required cut. Side clearance generally ranges from 3 to 10°. The front clearance is ground from the cutting edge and continues to the heel of the cutter bit. If too much clearance is ground on a tool, the cutting edge will wear rapidly, and will even break under heavy cutting loads. The front clearance generally ranges from 3° to 15°, depending on the nature of the work and the height at which the tool is set.

Cutting Tools for Nonferrous Metals. The ease with which nonferrous materials such as aluminum, bronze, copper, and plastics may be machined varies. Often these materials require special cutting tools. Table II lists the cutting speeds for these as well as some other metals.

Grinding Lathe Tools

The correct grinding of cutter bits is one of the most important steps in the machining of metal. The cutter bit must be ground correctly for the particular metal being machined and it must have a keen edge in order to work smoothly and efficiently. Correct grinding of a tool bit will serve two purposes: (1) it permits the work to be done more easily and at maximum speed; and (2) the steel retains its cutting edge as long as possible. The various clearances and rake angles must be ground correctly and ground so as to turn the chip, which helps preserve the cutting edge. Clearance angles which are extreme reduce the cross section of metal near the cutting edge and prevent the heat generated by the cutting from being properly dissipated.

Cutting tools vary considerably in shape, depending on the various jobs for which they are ground; there are, however, certain basic principles governing their shape. If these principles are learned and applied, the grinding of tools is comparatively easy.

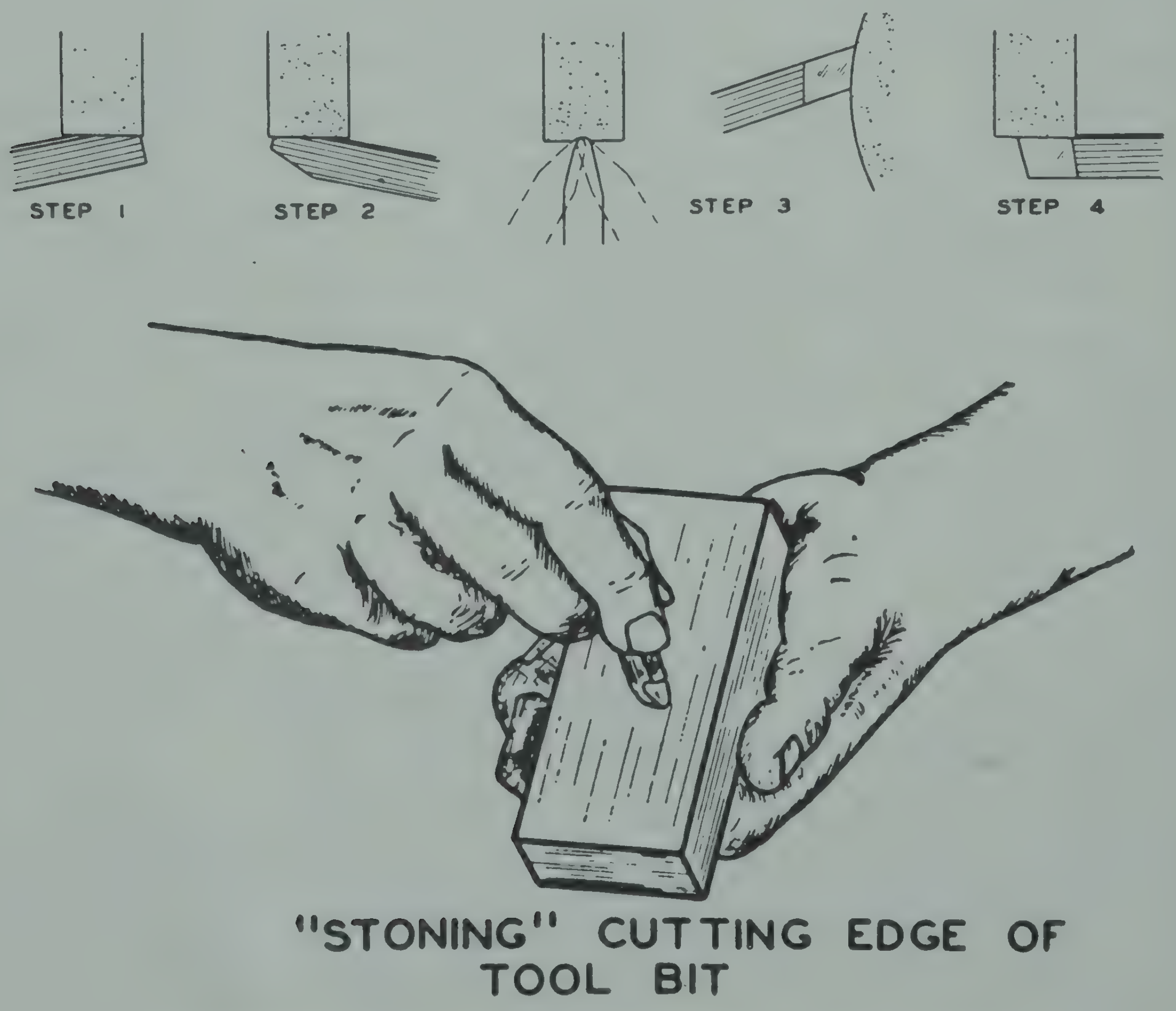


Figure 23. Grinding and honing lathe tools.

The cutting edge of a tool bit should be honed to provide a keener edge. This honing improves the quality of the cutter so that it produces a smoother finish surface on the work and in addition the cutting edge lasts longer. See Figure 23 for the steps in grinding and honing tool bits.

General Notes on Lathe Turning

Straight or Cylindrical Turning. This is the process of machining a piece of work in a lathe by moving a cutting tool longitudinally along the revolving piece in order to produce sides that are parallel to the axis of the work. Straight turning is divided into two stages, rough and finish turning. If a large amount of metal is to be removed, the roughing cuts are made as deep as the lathe, the work, and the tool will stand. Ordinarily work is rough turned to within $^1/_{32}$ in. of the finished size. Heavy feeds and slow speed are recommended for roughing cuts to help prevent undue wearing of the cutting edge. Finish cuts are taken to smooth and accurately size the work. The spindle speed is usually increased for a finish cut, while the amount of feed is decreased. Generally one finish cut is all that is necessary. The cutting edge of the tool bit is generally set at the height of the center, but for cutting soft steel and cast iron the tool may be set about $^1/_{32}$ in. above the center for each inch of work diameter. Care must be taken to provide sufficient clearance when a tool is set above center.

General Shop Notes.

1. A good lubricant for turning, boring, and milling aluminum can be made from equal parts of lard oil and kerosene. Kerosine alone may also be used.

2. When hard steel (not hardened steel) is being drilled in the lathe, it should be run slowly and the tool lubricated with turpentine or turpentine and spirits of camphor.

3. Red lead and graphite are good lubricants for the tool center.

4. On heavy work the countersink for the center should be as large as possible without making the job unsightly.

5. The lathe bed must be level, and should not be bolted to

the floor unless it has been properly leveled by means of shims.

6. Squared ends and a uniform depth of lathe center holes are desirable for accurate work.

7. A small square of mica with a tin rim makes a good chip guard in turning brass. A wire spring clip should be fastened to the metal rim so that the guard may be quickly fastened or detached from the tool post.

8. A $^1/_{32}$-in. slot made on the top of the tail center, running from the point to a little beyond the large part of the conical end, will permit the center to be oiled without slacking it away.

9. The lathe center should be ground in place if possible and well cared for. It should also be marked and always put in place in the same position.

10. The lathe center should not be used for a center punch.

11. A shaft should not be straightened between centers by hammering on it. The centers are likely not to run true afterwards.

12. A gage or caliper should not be used on a shaft while it is running.

13. A lathe tool should not be set below centers for external work.

14. A lathe should not be run if the lathe center is squealing. It should be stopped and the center lubricated.

15. The lathe tool should not be run into the face plate.

Filing and Polishing

Filing on a lathe is primarily a finishing operation intended to remove tool marks on turned surfaces and reduce diameters by small amounts. Filing is performed at higher spindle speeds than those used for turning purposes.

Polishing on a lathe is performed with abrasive cloth to produce a high finish. As in filing the work is operated at high spindle speeds and the coarseness of the abrasive cloth determines the degree of smoothness that can be obtained.

Hardening and Tempering Lathe Tools

Forged lathe tools which have been used for some time should be reforged, hardened, and tempered. If this is done carefully, the tool will be as good as new.

A lathe tool made of carbon tool steel is hardened by heating the end of the tool to a bright cherry red for a distance of 1 in. back of the cutting edge. The point is then immersed to a depth of about 1½ in. in cold water, but the shank should not be cooled. When the cutting edge is cool, the tool is removed from the water, and the edge is polished with emery cloth and wiped with an oily rag. This procedure hardens the tool. As the heat from the shank passes into the point, it discolors it, thus indicating the amount the temper was drawn.

Note: Carbon tool steel may be distinguished from high-speed steel by the nature of the sparks given off when the metal is touched against a revolving grinding wheel. Carbon steel gives off a shower of bright yellow sparks while high-speed steel gives off dark red sparks.

The same hardening method just described will harden and temper any tool made of carbon tool steel. Wood cutting tools, taps, and dies should be drawn to a dark straw color. Cold chisels, hatchets, screw drivers, etc., should be drawn to a brown-yellow, while springs should be drawn to a dark purple.

Casehardening

Machine steel is casehardened by heating it to a cherry-red and then applying cyanide of potassium to the section to be casehardened. The cyanide will dissolve slowly and is absorbed by the steel. After time is allowed for the steel to receive a thorough coating of cyanide, the steel is heated slowly in the fire for about 1 minute to be sure the cyanide is thoroughly absorbed by the steel, after which the steel is quenched in cold water.

Annealing Tool Steel

Carbon steel may be annealed by being heated slowly and evenly to a cherry red and then placed in a box of lime or ashes to cool slowly. The steel should be completely covered during cooling, and when it has cooled to room temperature, it is ready for machining.

Annealing Brass

Brass which has become work-hardened should be annealed by being heated to a dull red and plunged into cold water. Care must be taken not to overheat the brass during this operation.

SPOT WELDING OF METALS

Spot welding is the process in which two pieces of metal are joined together by an electric current which is made to flow through the point where the two metals touch. The metals are heated above their melting point by the electric current and are forced together by applied pressure. The rest of the material remains relatively cool owing to the conduction of heat through the electrodes.

Temperatures. The temperatures which are obtained in spot welding two pieces of metal depend on several factors such as:

1. The electrical and thermal conductivity of the metals being welded.
2. Size and shape of the pieces of metal.
3. Contact resistance between the pieces of metal themselves and between the metal and the electrode tips.
4. Specific heat and heat of fusion.
5. The electrodes. These must also be considered since the electrical and thermal conductivity, the specific heat, size, shape, and type of electrodes (whether solid or water-cooled) are factors pertaining to the temperature obtained in spot welding.

Variables in Obtaining Strong Welds. 1. There are variables in the spot-welding machine to be considered. These are the welding voltage at the electrical tips, the internal electrical resistance of the welder, the duration of the current flow, and the pressure exerted by the electrodes during the welding.

2. Other factors which must be considered in obtaining a strong finished seal are the inherent strength of the materials after the heat treatment induced by the spot-welding process, the cleanliness of the pieces being welded, the degree to which the metals wet or alloy, the strength of the alloy formed during the welding, and the size of the weld.

The strength of the finished weld depends on how well all these factors are adjusted together.

Technique

The pressure between the electrodes must be regulated according to the material being welded. Obviously considerably more pressure will be required to weld together two pieces of steel than two thin pieces of platinum. Too much pressure between the electrodes in welding the latter would cause a hole to be punched in the metal, or the metal to flash. (Flashing occurs when the metals being welded melt and produce an arc. This not only ruins the pieces being welded but spoils the electrodes.)

Electrodes. 1. For making welds with most high-resistance materials, cone-shaped copper electrodes are satisfactory. The tips stay relatively cool and so do not alloy with the material being welded.

2. For welding metals with high thermal and electrical conductivity, such as aluminum and copper, tungsten electrodes must be used. So much more heating current is required to make these welds that the electrode tips become hot and weld to the material as well as and possibly better than the two pieces of metal weld to each other. Even with tungsten tips welds between metals of high conductivity are difficult. This difficulty can be partly overcome by making the contact between material and electrodes large while the spot of contact between the metals themselves is kept small.

3. Welds between a strip of a metal of high conductivity and one of low conductivity also present a problem. Gillette[1] reported that the use of dissimilar electrodes to balance the unequal heating in the metals being welded would facilitate obtaining satisfactory welds. The unequal heating of copper and Nichrome (and similar high- and low-conductivity metals) can also be partially balanced by using a small electrode tip against the copper and a large electrode tip against the Nichrome.

4. Dissimilar tips may be used advantageously when the weld is being made between metals with different melting points or

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[1] Gillette, R. T., *Gen. Elec. Rev.*, **30**, 443 (1927).

specific heats. Dissimilar heating may sometimes be balanced by using metals of different thicknesses. For example, thin copper may be welded to a thick Nichrome strips easier than to strips of equal thickness.

Cooling Electrode Tips. Some spot welders are equipped with water-cooled electrodes.

Harmful Effects Accompanying Spot Welding. The local heating during spot welding has a harmful effect on some metals. Zinc is recrystallized and made brittle; iron is slightly oxidized; zinc is partly distilled from brass; and duraluminum and tool steel are given an undesirable heat treatment. These undesirable effects can be reduced by making the welds under the surface of a cooling liquid. Pfeiffer[2] found that welds made under carbon tetrachloride kept some alloys from becoming brittle. Iron may also be spot-welded under water without becoming oxidized.

Cleaning Metals To Be Welded. It is important that the metals be thoroughly cleaned, since even the best technique is useless with greasy or dirty materials. Scale and dirt make a poor electrical contact, which prevents the proper flow of electrical current. Should the welding voltage be increased and break through the contamination to form a weld, the dirt and scale will mix in the weld material, making the weld porous and brittle.

Welding Characteristics of Metals. Two hundred and fifty combinations of metals which can and cannot be successfully welded were listed by Ferguson[3] and are shown in Table V. It has been noticed by the Bell Telephone Laboratories,[4] where this research in welding was carried out, that whereas all the combinations of metals shown to be weldable are actually weldable, they are not necessarily weldable on a mass production basis. In fact, in many cases considerable pains were necessary to accomplish the welds. The voltages listed in Table VI for spot-welding various metals apply to a 60 K No. 2 Gibb welder under prescribed conditions and will not apply to other welders, but are

[2] Pfeiffer, I., *Am. Welding Soc. J.*, **10**, 5 (1931).

[3] Ferguson, L., *Welding Eng.*, **18**, 14, July (1933) *ibid.*, **18**, 20 (1933).

[4] Bell Telephone Laboratories, 463 West St., New York 14, New York, personal communication.

TABLE V. Characteristics of Spotwelding

■ GOOD WELD
• COMPLETELY MISSIBLE BUT BRITTLE WELD
⊗ POOR WELD
○ NO WELD
BLANK SPACE-COMBINATION NOT TRIED

	IRON	STAINLESS IRON	ALLEGHENY METAL	COBALT STEEL	NICKEL	NICHROME	MONEL METAL	NICKEL SILVER	BRASS	BRONZE	MANGANIN	EVERDUR	COPPER	ALUMINUM	MAGNESIUM	MOLYBDENUM	LEAD	TIN	CADMIUM	ZINC	GALVANIZED IRON *	TIN PLATE *	CHROMIUM PLATED ST. *	NICKEL PLATED BRASS *
IRON	■																							
STAINLESS IRON	■	■																						
ALLEGHENY METAL	■	■	■																					
COBALT STEEL	■	■	■	■																				
NICKEL	■	■	■	■	■																			
NICHROME	■	■	■	■	■	■																		
MONEL METAL	■	■	■	■	■	■	■																	
NICKEL SILVER	■	■	■	■	■	■	■	■																
BRASS	■	■	■	■	■	■	■	■	■															
BRONZE	■	■	■	■	■	■	■	■	■	■														
MANGANIN	■	■	■	■	■	■	■	■	■	■	■													
EVERDUR	■	■	■	■	■	■	■	■	■	■	■	■												
COPPER	■	■	■	■	■	■	■	■	■	■	■	■	■											
ALUMINUM	■	■	■	⊗	○	•	⊗	⊗	•	⊗	•	•	•	■										
MAGNESIUM										⊗				■	■									
MOLYBDENUM	■	■	■	■	■	■	■	■	○	○	■		○	⊗		■								
LEAD							⊗	■	○	○	■	■					■							
TIN	○	○			⊗	○	■	■	■	■	■	■	■	○			■	■						
CADMIUM	■				⊗	○	■	■	■	■	■	■	■	○			■	■	■					
ZINC	⊗				⊗	⊗	•	•	•	•	•	•	•	■			⊗	⊗	⊗	•				
* GALVANIZED IRON	■	■	■	■	■	■	■	■	■	■	■	■	■	⊗			⊗	■	■	⊗	■			
* TIN PLATE	■	■	■	■	■	■	■	■	■	■	■	■	■	■			■	■	■	⊗	■	■		
* CHROMIUM PLATED ST.	■	■	■	■	■	■	■	■	⊗	⊗	■		■	○							■	■	■	
* NICKEL PLATED BRASS	■	■	■	■	■	■	■	■	■	■	■	■	■	⊗		■	○	■	■	⊗	■	■	⊗	■

* IN THE COURSE OF SPOT WELDING COATED MATERIALS, THE COATINGS FREQUENTLY DISSOLVE IN THE OTHER METALS PRESENT OR BURN AWAY

TABLE VI. Voltages Necessary for Spotwelding

	IRON	STAINLESS IRON	ALLEGHENY METAL	COBALT STEEL	NICKEL	NICHROME	MONEL METAL	NICKEL SILVER	BRASS	BRONZE	MANGANIN	EVERDUR	COPPER	ALUMINUM	MAGNESIUM	MOLYBDENUM	LEAD	TIN	CADMIUM	ZINC	GALVANIZED IRON *	TIN PLATE *	CHROMIUM PLATED ST. *	NICKEL PLATED BRASS *
IRON	1.5																							
STAINLESS IRON	1.5	1.93																						
ALLEGHENY METAL	1.75	1.93	2.28																					
COBALT STEEL	1.5	1.75	2.1	1.75																				
NICKEL	1.5	1.75	1.89	1.75	1.78																			
NICHROME	1.5	1.5	1.5	1.75	1.5	1.5																		
MONEL METAL	1.7	1.78	2.0	1.93	1.89	1.5	1.89																	
NICKEL SILVER	1.7	1.89	2.0	1.78	1.93	1.5	1.89	2.2																
BRASS	1.7	2.68	2.2	1.93	2.1	1.93	1.89	2.1	2.55															
BRONZE	1.7	2.28	2.1	2.0	2.63	1.75	2.0	2.55	2.1	2.9														
MANGANIN	1.5	1.7	1.75	1.5	1.89	1.5	1.7	1.89	1.89	1.89	1.5													
EVERDUR	1.5	1.7	2.1	1.78	1.93	1.5	1.89	2.1	2.68	2.28		1.34												
COPPER	1.75	2.2	2.1	1.93	2.43	2.1	2.1	2.1	2.55	3.78	2.2	3.26	2.63											
ALUMINUM	1.45	1.5	1.5	1.5	3.55	1.7	2.9	2.43	2.28	2.28	1.34	2.28	1.5	2.55										
MAGNESIUM										4.4				3.26	3.02									
MOLYBDENUM	1.5	2.1	1.7	1.75	1.75	1.93	1.89	1.93	3.5	1.5	1.34		2.9	2.0		1.75								
LEAD							1.7	1.5	1.5	1.5	1.5	1.5					1.28							
TIN	1.22	1.14			1.14	1.14	1.28	1.28	1.34	1.34	1.34	1.75	1.5	1.63			1.89	1.75						
CADMIUM	1.34				1.75	1.34	1.89	2.0	2.2	2.1	1.45	2.55	1.75	1.14			2.1	1.75	3.55					
ZINC	1.05				1.22	1.34	1.63	1.63	1.93	1.75	1.63		1.78	1.14			1.78	1.34	1.75	1.75				
* GALVANIZED IRON	1.45	1.5	1.7	1.5	1.7	1.34	1.5	1.7	2.1	2.0	1.63	1.63	2.55	1.45			1.5	1.5	1.7	1.7	1.34			
* TIN PLATE	1.5	1.7	2.0	1.7	1.93	1.5	1.89	2.1	2.1	1.93	1.75	1.89	2.2	1.5			1.5	1.5	1.75	1.7	1.7	2.1		
* CHROMIUM PLATED ST.	1.5	1.7	1.7	1.7	1.75	1.5	1.7	1.75	2.1	2.2	1.5		2.28	2.1							1.7	1.75	1.89	
* NICKEL PLATED BRASS	1.7	2.43	2.1	1.93	2.0	1.89	1.89	2.1	2.1	2.2	1.75	2.43	2.43	2.1		1.93	1.5	1.63	2.2	2.0	1.93	2.1	2.55	2.2

* THE POTENTIALS GIVEN FOR THESE COATED MATERIALS MAY BE SUFFICIENT TO DISSOLVE THE COATINGS IN THE OTHER METALS PRESENT OR TO BURN THEM AWAY

given as a comparison of the voltages necessary to make different welds. Each spot welder has some method of regulating voltages and by using the figures in Table VI as a guide, the current ratio between one weld and another may be estimated.

SOLDERING

Solders can be divided into two kinds, soft solders, which melt below 300° C., and hard solders, which do not generally melt below 650° C. An important ingredient in successful soldering is some form of a flux. The flux must be fluid at below the melting point of the solder so that it can flow over the surface of the metal and clean it. This also permits it to avoid being trapped in the solder and weakening the joint.

Thorough cleaning of the metal surfaces is essential for successful soldering.

Soft Solders

Table VII lists the composition of some soft solders, their fluxes, and the metals which they are useful for soldering. Successful soft solder joints may usually be more easily obtained if the individual parts are pretinned before the pieces are assembled.

The temperature of soft solder should not be raised above that necessary to cause the solder to flow, in order to avoid excessive oxidation. Soft soldering is generally done either by heating the metal with an open flame or by using an electrical soldering iron. For large or complicated work requiring several solder joints, the best results will be achieved using a combination of flame and electric soldering iron for heating. A soft gas-air flame is played over the work to bring the entire surface to between 125° to 150° C., after which a soldering iron is used to melt the solder and make the actual seal. The amount of solder added and the direction of flow are more easily controlled with the soldering iron than by a flame alone.

When an open flame is used as the sole means of heating the metal for soft solder seals, the best results will generally be

TABLE VII. Soft Solders and Fluxes for Various Metals

Metals to be soldered	Flux	Composition of soft solder					
		Tin	Lead	Zinc	Aluminum	Phosphor tin	Bismuth
Aluminum	Stearin	70		25	3	2	
Brass	Chloride of zinc, rosin, or chloride of ammonia	66	34				
Gun metal		63	37				
Copper		60	40				
Lead	Tallow or rosin	33	67				
Block tin	Chloride of zinc	99	01				
Tinned steel	Chloride of zinc or rosin	64	36				
Galvanized steel	Hydrochloric acid	58	42				
Zinc	Hydrochloric acid	55	45				
Pewter	Gallipoli oil	25	25				50
Iron and steel	Chloride of ammonia	50	50				
Gold	Chloride of zinc	67	33				
Silver	Chloride of zinc	67	33				
Bismuth	Chloride of zinc	33	33				34

achieved when the flame is not played directly on the surfaces being soldered. In some cases, however, after the seal is completed the appearance of the seal may be improved by spot-heating. This may be done by playing a flame directly on a section of the seal, causing the solder to melt and flow smoothly at this point. Care should be exercised during any spot heating to avoid heating the whole solder joint.

Soft solder joints in general are not as strong as hard solder joints and therefore should be used only when mechanical strength is not required or when the work would be injured by the high temperature required for hard soldering.

Hard Soldering

Table VIII lists the metals, fluxes and compositions of the hard solders that can be used. One of the best hard solders is a commercial Easy Flow solder made by Handy and Harmon Co.

TABLE VIII. Hard Solders and Fluxes for Various Metals

Metals to be soldered	Flux	Hard solder			
		Copper	Zinc	Silver	Gold
Brass, soft	Borax	22	78		
Brass, hard	Borax	45	55		
Copper	Borax	50	50		
Gold	Borax	22		11	67
Silver	Borax	20	10	70	
Cast iron	Cuprous oxide	55	45		
Iron and steel	Borax	64	36		

As will be noted from the table, most of the hard solders are alloys of copper and zinc. An easily fusible hard solder may be made of one part copper to two parts of zinc; this, however, makes a joint that will be weaker than when an alloy more difficult to melt is used.

The heat required for hard soldering is obtained with a gas-air or gas-oxygen flame, depending on the size of the work being heated. Owing to the fact that silver solder flows more readily than soft solder, the metal parts to be fitted must be machined to a clearance of only a few thousandths of an inch. After being assembled and preheated, the regions to be soldered are painted with the Easy Flow flux. The extent of the solder joint is determined by painting with the flux, since the solder will wet the surface of the metal only to the extent that it has been painted with flux. When the joints fit together smoothly, only a thin film of silver solder will be necessary to make a strong vacuum-tight seal. Additional solder makes for a joint which is usually poor in appearance. The excess solder may be removed by heating the joint until the solder is fluid and then wiping the joint with a damp cloth or pad of steel wool.

Aluminum Solder

Aluminum may be soldered if the parts to be soldered are previously tinned with an alloy of aluminum and tin which consists of five parts tin and one part aluminum. Soldering is done with a soldering iron in which the copper tip is replaced with a pure aluminum tip, using the solder described in Table VII.

Glass Solders

Glass may be soldered to glass or a glass-to-metal solder seal may be made using one of the three following solders.

1. Tin is melted and enough copper is added to make an alloy of 95 % tin and 5 % copper. This alloy should be stirred constantly while it is melting. The solder may be made more or less hard by adding ½ % to 1 % of zinc.

2. An alloy of 95 % tin and 5 % zinc will melt at 392° F. and can be firmly united to glass.

3. An alloy of 90 % tin and 10 % aluminum melts at 734° F. and also adheres firmly to glass.

With either of these last two alloys glass may be soldered as easily as metal. This may be done two ways. (1) The glass should be heated in a furnace until a stick of the solder will melt when rubbed over the surface of the glass. The solder is then spread evenly over the surface by means of a roll of paper or strip of aluminum. The second piece of glass is pressed firmly in place and the two allowed to cool. (2) A common soldering iron is heated and the soldering done as usual, without the use of a flux. Care should be exercised that neither the glass nor the soldering iron be heated to a temperature above the melting point of the solder, to prevent it from becoming oxidized. Should the solder become oxidized it will not adhere to the glass and it should be removed completely and the solder seal started again.

Tinning Glass and Ceramics Preparatory to Soldering

A new technique of soldering such materials as glass, ceramics, aluminum, and stainless steel, as well as any combination of these materials, has been reported by McGuire.[5] The usual soldering materials are used in addition to a hand grinder with an abrasive wheel which has been loaded with a soft solder alloy.

Loading Abrasive Wheel. To load these abrasive wheels the grinder should be turned on and the wheel (preheated by grinding metal or applying heat) brought to bear on soft solder such as Wood's metal or a 60–40 lead-tin solder. Under the proper conditions the soft solder melts and flows onto the surface

[5] McGuire, J. C., *Electronics*, **29**, 243 (1956).

of the wheel. This loading process should be repeated two or three times to build up a heavy coating of alloy on the wheel. According to McGuire there seemed to be a definite conditioning period for new grinding wheels; however, after a good deposit had been built up it was easy to renew the layer after each tinning use. Again according to McGuire, while the size of the wheel was not critical, a wheel ¼ in. in diameter and ½ in. in length was found to produce the best results.

Tinning Procedure. In tinning the surfaces to be soldered, the solder loaded wheel should be applied with the same pressure used in grinding. The heat of friction will melt the soft solder sufficiently for it to flow onto the freshly abraded surface. When possible, the work should be warmed to the melting temperature of the tinning alloy (about 80° C) as a preliminary step to tinning. This may be done by applying a hot soldering iron to the area when metals are being tinned, or by using a flame when working with glass or ceramics.

Failure to deposit a satisfactory tinned coating is an indication that the wheel was too cold when it was loaded, or that it was not completely loaded.

If the rotational speed of the grinder is too fast, Wood's metal will be laid down in a black deposit on glass and solder will not adhere to it. In this case the speed of the grinder should be reduced and grinding continued until a bright, shiny surface appears. A black spot will also appear if the surface of the glass has oil or grease on it.

Soldering Procedure. When soldering metal, the metal should be heated first by applying the hot iron to some adjacent part which is not tinned. When the metal is hot, solder should be applied directly to the tinned surface or applied to the iron and the drop of hot solder brought down to the tinned area without bringing the iron into direct contact with the base metal. This is especially important when soldering titanium, niobium, and tantalum.

When glass or ceramics are being soldered, it will be necessary to use a flame or oven heating to heat the surfaces sufficiently for the solder to flow and form a seal.

Choice of Solders. The best tinning alloy for such materials as soft glass Pyrex, and ceramic was found to be Wood's metal and a 50–50 indium-tin[6] solder. This alloy bonds firmly enough to the glass so that ordinary higher melting solder may be used to form a surprisingly strong seal.

Many of the low melting alloys ranging from 40 to 140° C in melting point were reported to have been satisfactory for the tinning operation.

Solders used included 40–60 and 50–50 lead-tin solders as well as the 50–50 tin-indium and 50–50 lead-indium solders.

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[6] Indium-tin, commercially known as "Cerroseal", is available from Pick Steel Co., 4436 Long Beach Ave, Los Angeles, California.

XV

ELECTRICAL HEATERS

Since in many cases special research apparatus is designed with built-in electrical heaters, the glassblower must be able to design heaters of specific size and wattage.

DESIGNING COIL UNITS

The following data are offered merely as a quick short-cut in designing heating elements made from Chromel wire and ribbon. Chromel wire, as made by the Hoskins Manufacturing Co.,[1] is an alloy of 80 % nickel and 20 % chromium, and has been produced commercially since 1909. In use, Chromel A wire has become the standard resistance alloy for electric heating appliances and industrial furnaces. It is highly satisfactory for all purposes where the heating element is operated above a bright red heat and exposed to the air. The fundamental laws upon which the following tables were calculated are outlined below.

Ohm's Law

Any electrical resistance type heating unit will operate on a.c. or d.c. and the following laws apply:

volts = ohms × amperes $E = R \times I$
amperes = volts ÷ ohms $I = E \div R$
ohms = volts ÷ amperes $R = E \div I$
watts = volts × amperes $W = E \times I$
watts = (volts)² ÷ ohms $W = E^2 \div R$
watts = (amperes)² × ohms $W = I^2 \times R$
ohms = watts ÷ (amperes)² $R = W \div I^2$

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[1] Lawton Avenue and Buchanan Street, Detroit 8, Michigan.

Cold and Hot Resistance

The term "cold resistance" refers to the resistance of wire at room temperature just as it is taken from the spool. "Hot resistance" is the resistance of the wire when it is in operation as a heating element. The hot resistance is higher than the cold resistance by an amount which varies with the temperature, and which is shown in terms of percentage rise in resistance tables.

The ohm, as calculated in the preceding formula, is the resistance necessary for a given wattage in a device when it is at the operating temperature. For the average heater the hot resistance of Chromel A will be 7 % more than its cold resistance. When Chromel C is used, the corresponding value is 12 %, and when Chromel D is used, the figure is 22 %. It is recommended by Hoskins Manufacturing Co. that whenever possible a test be made on a finished heater to determine the proper allowance for increase in resistance. This is particularly true when a large number of one-dimension heaters are being made. Even for the same grade in wire, the resistance may not be the same for all designs of heaters. For example, a 500-watt element on a 115-volt circuit will draw 4.35 amperes when heated and the hot resistance will be $R = E \div I$ or 26.5 ohms. If the element is made from Chromel A, the hot resistance will be 7 % higher than the cold resistance; therefore, the cold resistance will be $26.5 \div 1.07$ or 24.7 ohms.

Heating Element Design

General Notes on Heating Elements. 1. Welded, brazed, or spot-welded joints between Chromel wire and terminals are the most satisfactory. Pressed or pressure joints are usually satisfactory, but they should be thoroughly tested before being sealed into an apparatus.

2. If a pressure joint is made, care should be exercised to avoid loose contact trouble due to stretching or yielding of the clamping device. It is better to place the alloy wire between two nuts than to place it under only one nut.

3. Where extremely accurate wattage is important, the finished element should be tested to determine the proper allowance for rise in resistance with temperature.

4. Elements should be designed so they are free to expand or contract. If an element must be anchored in any way, it should be tested to be sure that warpage or creeping is not going to cause trouble.

5. Heating elements should be mounted in such a way that the heat may "flow" as easily as possible into the work to be heated.

6. When necessary, "half gage sizes" may be calculated by multiplying the smaller AWG diameter by 1.06.

7. If the heating element is for use with refractories, it is necessary that the refractory be free from potassium and sodium, since these elements are especially harmful on direct-current heating units.

8. It is necessary to remember that for a given element design, the wire length is reduced or the wire diameter increased if more power is to be drawn.

9. A 1 % excess of voltage causes a 2 % excess of wattage.

10. It is safe to stretch a coil from 1½ to 4 times its close-wound diameter.

11. A heavier wire will probably give a longer life when operated as an exposed heating unit. When the heat may be easily dissipated from the wire, as in a heater immersed in liquid, a smaller wire may be used safely.

Notes on Heating Element Design. 1. A helical coil whose ratio of "arbor size to wire size" is over 10 should not be designed unless it is absolutely necessary. Such coils are flimsy and difficult to make.

2. A hot wire should not be exposed to sulfur gases, especially in a reducing atmosphere. If this is necessary, the lower nickel alloy, Chromel D, may be more durable.

3. An element should not be designed for 115 volts and then overloaded with 230 volts. From the beginning the design should be based on the requirements of 230 volts. It is more difficult to work out a 230-volt heating unit for smaller devices so 115 volts should be used for these whenever possible.

4. The hot wire should not be exposed to dripping oil, oil-spray, or contaminating gases which may evolve from some grades of asbestos or low-temperature magnesia insulation.

5. A pressure joint should not be made between the alloy wire and brass, if the brass temperature is to exceed 300° F. The zinc in the brass will oxidize and form an insulating film.

6. If twisted "tails" are used, the loop end should not be cut off because current may flow from the terminal into the loose strand and then into the element and cause trouble.

7. If the wire is to be wrapped around the terminal, it should be wrapped in a clockwise direction. The wire should not be lapped because the pressure may partially cut through it.

8. In taking the wire off the spool, the end should not fly loose and get under a turn of the wire, since this forms a loose knot and often causes a tangle.

9. In winding wire into coils or onto forms, excessive tension should not be used on the wire. This may cause the wire to pull down into the unspooled portion and result in either a break, stretch, or tangle.

Estimating Coiled Heating Elements. The data from Tables I, II, and III may be used in the following manner: When the length of the groove or space available for the element is known, and the approximate arbor size for the coiled wire is decided, the required wattage should be calculated or estimated. From one of the five sizes of wire listed under voltage, the size that may best fit the desired application is selected, with the provision that a coil will usually be stretched from 1½ to 4 times its close-wound length. Table I will be found useful for estimating the stretched length of helical coils of various sizes of wire and diameter of arbor.

Example: A 700-watt, 115-volt open-type electric muffle element is desired. The groove containing the coil is 37 in. long and large enough for a wire wound around a $^5/_{42}$-in. arbor. The O.D. of a coil will be approximately equal to the diameter of the arbor, plus two times the diameter of the wire, plus 0.005 in.

Under the 700-watt column (Table I) it will be seen that the element will draw, when hot, 6.09 amperes and the cold resistance will be 17.65 ohms. Any of the five sizes of Chromel A, 18 to 22 inclusive, may be chosen. With the use of 20 gage, since this is the middle size and will heat to about 1650° as an open-

TABLE I. Coiled Heating Element Design — 115 volts a.c.-d.c.

CHROMEL-A • COILED HEATING ELEMENT DESIGN DATA • *115 Volts—AC or DC*

To apply these figures to Chromel-C, reduce the lengths 7%.

For use, it is necessary to stretch coils from 1½ to 4 times their close-wound length.

Watts →		20	40	60	80	100	150	200	300	400	500	600	700	800	900	1000	1100	1200	1300	1400	1500		
Amperes →		.174	.348	.522	.695	.87	1.31	1.74	2.61	3.48	4.35	5.22	6.09	6.96	7.83	8.70	9.56	10.44	11.30	12.18	13.05		
Hot Ohms* →		661	331	221	165.5	132.3	88.3	66.2	44.2	33.1	26.5	22.1	18.88	16.54	14.69	13.22	12.02	11.02	10.16	9.44	8.81		
Cold Ohms →		618	309	206	154.5	123.5	82.4	61.8	41.2	30.9	24.7	20.6	17.65	15.44	13.72	12.35	11.23	10.30	9.50	8.82	8.23		
A.W.G. or B. & S.	Ohms per Ft. @ 68° F.	FEET OF STRAIGHT WIRE TO MAKE HELICAL COILS FOR ABOVE WATTAGES																				A.W.G. or B. & S.	Dia. Inches
13	.1255																				65.6	13	.072
14	.1588																		59.8	55.5	51.8	14	.064
15	.200																56.2	51.5	47.5	44.1	41.2	15	.057
16	.250														54.9	49.3	44.9	41.2	38.0	35.3	32.9	16	.051
17	.321													48.1	42.8	38.5	35.0	32.1	29.6	27.5	25.6	17	.045
18	.406												43.5	38.1	33.8	30.4	27.7	25.4	23.4	21.7		18	.040
19	.502											41.1	35.2	30.8	27.3	24.6	22.4	20.5				19	.036
20	.635										38.9	32.4	27.8	24.3	21.6	19.0						20	.032
21	.800										30.9	25.8	22.1	19.3								21	.0285
22	1.017									30.4	24.3	20.3	17.4									22	.0253
23	1.272									24.3	19.4	16.2										23	.0226
24	1.609								25.6	19.2	15.4											24	.0201
25	2.03								20.3	15.2												25	.0179
26	2.57							24.1	16.0	12.0												26	.0159
27	3.23						25.5	19.1	12.8													27	.0142
28	4.09					30.2	20.2	15.1	10.1													28	.0126
29	5.09				30.4	24.3	16.2	12.1														29	.0113
30	6.50				23.8	19.0	12.7	9.5														30	.0100
31	8.21			25.1	18.8	15.1	10.0															31	.0089
32	10.16			20.3	15.2	12.2																32	.0080
33	12.90		24.0	16.0	12.0																	33	.0071
34	16.37		18.9	12.6																		34	.0063
35	20.7		14.9	10.0																		35	.0056
36	26.0	23.8	11.9																			36	.0050
37	32.1	19.3	9.6																			37	.0045
38	40.6	15.2																				38	.0040
39	53.1	11.6																				39	.0035
40	67.6	9.1																				40	.0031

The middle size in each group will operate at about 1200° F. in open air. In a device the coil temperature will be higher. The heavier wire will usually make a more satisfactory heating element.

TABLE II. Coiled Heating Element Design — 230 volts a.c.-d.c.

CHROMEL-A • COILED HEATING ELEMENT DESIGN DATA—*Continued*

230 Volts—AC or DC

	Watts →	40	80	100	150	200	300	400	500	600	700	800	900	1000	1200	1400	1600	1800	2000	2500	3000		
	Amperes →	.174	.348	435	.652	.87	1.31	1.74	2.17	2.61	3.04	3.48	3.91	4.35	5.22	6.09	6.96	7.83	8.70	10.88	13.05		
	Hot Ohms →	1323	662	529	353	265	176	132	105.8	88.2	75.6	66.1	55.8	52.9	44.1	37.8	33.1	29.4	26.4	21.2	17.6		
	Cold Ohms →	1237	618	494	330	247	165	124	98.9	82.4	70.7	61.8	55.0	49.4	41.2	35.3	30.9	27.5	24.7	19.8	16.5		
A.W.G. or B. & S.	Ohms per Ft. @ 68° F.	FEET OF STRAIGHT WIRE TO MAKE HELICAL COILS FOR GIVEN WATTAGES																				A.W.G. or B. & S.	Dia. Inches
13	.1255																				131.5	13	.072
14	.1588																			124.7	104.0	14	.064
15	.200																		123.4	99.0	82.5	15	.057
16	.250																	110.0	98.8	79.2	66.0	16	.051
17	.321																96.3	85.7	76.9	61.7	51.4	17	.045
18	.406															87.0	76.1	67.7	60.8	48.8		18	.040
19	.502														82.1	70.4	61.6	54.8	49.2			19	.036
20	.635													77.8	65.9	55.6	48.7	43.3				20	.032
21	.800												68.8	61.8	51.5	44.2	38.6					21	.0285
22	1.017											60.8	54.1	48.6	36.9	34.7						22	.0253
23	1.272										55.6	48.6	43.3	38.8	32.4							23	.0226
24	1.609									51.2	43.9	38.4	34.2	30.7								24	.0201
25	2.03								48.7	40.6	34.8	30.4	27.1									25	.0179
26	2.57							48.3	38.5	32.1	27.5	24.1										26	.0159
27	3.23						51.1	38.4	30.6	25.5	21.9											27	.0142
28	4.09					60.4	40.7	30.6	24.2	20.2												28	.0126
29	5.09					48.5	32.4	24.4	19.4													29	.0113
30	6.50				50.8	38.0	25.4	19.1														30	.0100
31	8.21				40.2	30.1	20.1															31	.0089
32	10.16			48.6	32.5	24.3																32	.0080
33	12.90		47.9	38.3	25.6	19.2																33	.0071
34	16.37		37.8	30.2	20.2																	34	.0063
35	20.7		29.9	23.9																		35	.0056
36	26.0	47.3	23.8	19.0																		36	.0050
37	32.1	38.5	19.3																			37	.0045
38	40.6	30.5																				38	.0040
39	53.1	23.3																				39	.0035
40	67.6	18.3																				40	.0031

TABLE III. Helical Coil Winding Data

CHROMEL-A • HELICAL COIL WINDING DATA

Lineal Feet and Cold Ohms per Inch of Helix

A. W. G. or B. & S.	Dia. Inches	ARBOR DIAMETER—INCHES																A. W. G or B. & S.	Ohms per Ft. @ 68° F. Straight Wire
		1/32		1/16		3/32		1/8		5/32		3/16		7/32		1/4			
		PER INCH OF CLOSE WOUND COIL																	
		Ft	Ohms	Ft.	Ohms	Ft.	Ohms	Ft.	Ohms	Ft.	Ohms.	Ft.	Ohms	Ft.	Ohms	Ft.	Ohms		
12	.081															1.09	.11	12	.0991
13	.072													1.08	.13	1.20	.15	13	.1255
14	.064											1.06	.17	1.19	.19	1.31	.21	14	.1588
15	.057											1.16	.23	1.30	.26	1.45	.29	15	.200
16	.051									1.12	.28	1.29	.32	1.41	.35	1.56	.39	16	.250
17	.045							1.02	.33	1.22	.39	1.43	.46	1.58	.51	1.76	.56	17	.321
18	.040							1.11	.45	1.35	.55	1.56	.63	1.76	.71	2.00	.81	18	.406
19	.036							1.21	.61	1.41	.74	1.72	.85	1.96	.98	2.22	1.11	19	.502
20	.032					1.06	.67	1.33	.84	1.64	1.04	1.91	1.21	2.19	1.39	2.50	1.59	20	.635
21	.0285					1.15	.93	1.46	1.17	1.79	1.44	2.11	1.69	2.46	1.97	2.80	2.24	21	.800
22	.0253					1.27	1.29	1.62	1.65	2.03	2.06	2.36	2.40	2.73	2.77	3.16	3.21	22	1.017
23	.0226					1.40	1.78	1.78	2.27	2.25	2.87	2.65	3.37	3.06	3.90	3.55	4.50	23	1.272
24	.0201			1.10	1.77	1.55	2.49	2.00	3.22	2.50	4.03	3.00	4.83	3.41	5.50	3.95	6.35	24	1.609
25	.0179			1.23	2.50	1.72	3.50	2.22	4.50	2.75	5.58	3.30	6.70	3.85	7.82			25	2.03
26	.0159			1.35	3.47	1.91	4.92	2.48	6.38	3.09	7.95	3.71	9.54					26	2.57
27	.0142			1.48	4.79	2.13	6.88	2.75	8.88	3.45	11.1							27	3.23
28	.0126			1.65	6.76	2.38	9.75	3.12	12.8	3.85	15.8							28	4.09
29	.0113	.95	4.85	1.83	9.32	2.64	13.5	3.45	17.6									29	5.09
30	.0100	1.02	6.63	2.00	13.0	2.95	19.2	3.84	24.9									30	6.50
31	.0089	1.12	9.14	2.25	18.5	3.29	27.0											31	8.21
32	.0080	1.25	12.7	2.49	25.4	3.67	37.3											32	10.16
33	.0071	1.33	17.2	2.73	35.2													33	12.90
34	.0063	1.47	24.1	3.05	50.0													34	16.37
35	.0056	1.65	34.1	3.42	70.8													35	20.7
36	.0050	1.84	47.9	3.80	98.8													36	26.5
37	.0045	2.04	65.4															37	32.1
38	.0040	2.25	91.6															38	40.6
39	.0035	2.48	132															39	53.1
40	.0031	2.76	187															40	67.6
	.00275	3.10	267															.00275	86.0
	.00250	3.54	368															.00250	104

—Ratio—

"I. D. of coil to Wire diameter" for coils shown ranges between $\frac{3}{1}$ and $\frac{12.5}{1}$

type unit, it will be seen that a coil of this size wire should be 27.8 linear feet in length.

Table III shows that 1 in. of close-wound helical coil of No. 20 gage on a $^5/_{32}$ in. arbor will contain 1.64 linear feet of wire. Therefore, since 27.8 linear feet is required in the heater, the close wound coil will be 27.8 ÷ 1.64 or 17 inches long. To make this coil fit in a 37 in. groove, it must be stretched 2¼ times its close-wound length.

With a heavier wire, for example, gage 19, Table I shows that 35.2 feet of this wire is required, which, wound on a $^5/_{32}$ in. arbor, equals 1.41 linear feet per inch of close-wound helical coil. This coil therefore will be 35.2 ÷ 1.41 or 25 in. and in order to fit, it must be stretched 1.5 times its close-wound length. From this it may be seen that either 20 gage or 19 gage wire may be used.

Data for Estimating Requirements for Coiled Heating Elements. The accompanying tables are intended to serve only as guides. Specific engineering advice on heating element design may be obtained from the Hoskins Manufacturing Co.

Table I gives the data necessary to design a coiled heating element for 115 volts a.c. or d.c. In using these figures it should be remembered that the hot resistance is 7 % higher than the cold resistance.

For each volt above 115 (up to 130) these lengths should be increased by 1.9 %, and for each volt below 115 (down to 100) these lengths may be decreased by 1.9 %.

Table II gives the data necessary to design a coiled heating unit for 230 volts a.c. or d.c. These lengths should be increased by 1.9 % for each 2 volts above 230 (up to 260) and decreased by 1.9 % for every 2 volts below 230 (down to 200).

Table III gives data for helical coil winding. The elongation of the wire and the expansion of the turns vary with the tension of winding, with the size of the wire, and with the ratio of I.D. of coil to wire diameter. The values in Table III are for estimating only; however, the ohms per inch may be used for Chromel A and C with reasonable accuracy. Coils are generally stretched from 1½ to 4 times the close-wound length.

Table IV gives the current carrying capacity of Chromel A

TABLE IV. Current-Carrying Capacity of Straight Wire

CURRENT CARRYING CAPACITY OF

CHROMEL-A and C STRAIGHT WIRE

Approximate Amperes to heat a Straight, Oxidized wire in quiet air to given temperatures:

Degrees Fahr.		400	600	800	1000	1200	1400	1600	1800	2000
Degrees Cent.		205	315	427	538	649	760	871	982	1093
A. W. G. or B. & S.	Inches Diam.	Amperes								
1	.289	78	108	139	176	219	268	322	377	434
2	.258	65	92	117	148	185	227	271	320	366
3	.229	55	77	98	124	156	190	228	267	306
4	.204	46	65	83	104	130	159	190	224	258
5	.182	39	55	70	88	107	132	160	188	217
6	.162	32.5	46	58	73	91	112	135	158	181
7	.144	27.4	38	49	62	77	94	114	134	153
8	.128	22.4	32.0	41	52	65	79	95	111	128
9	.114	18.8	26.8	34.5	44	55	67	80	94	108
10	.102	16.2	23.3	29.7	37.5	46	56	68	80	92
11	.091	13.8	19.2	24.8	31.5	39.0	48	57	67	78
12	.081	11.6	16.1	20.8	26.5	33.5	40.8	48	56	65
13	.072	9.8	13.6	17.6	22.5	28.2	34.2	41.0	48	55
14	.064	8.4	11.6	15.0	18.8	23.5	29.0	34.6	40.5	46
15	.057	7.2	10.0	12.8	16.1	20.0	24.5	29.4	34.3	39.2
16	.051	6.4	8.7	10.9	13.7	17.0	20.9	25.1	29.4	33.6
17	.045	5.5	7.5	9.5	11.7	14.5	17.6	21.1	24.6	28.1
18	.040	4.8	6.5	8.2	10.1	12.2	14.8	17.7	20.7	23.7
19	.036	4.3	5.8	7.2	8.7	10.6	12.7	15.2	17.8	20.5
20	.032	3.8	5.1	6.3	7.6	9.1	11.0	13.0	15.2	17.5
21	.0285	3.3	4.3	5.3	6.5	7.8	9.4	11.0	12.9	14.8
22	.0253	2.9	3.7	4.5	5.6	6.8	8.2	9.6	11.0	12.5
23	.0226	2.58	3.3	4.0	4.9	5.9	7.0	8.3	9.6	11.0
24	.0201	2.21	2.9	3.4	4.2	5.1	6.0	7.1	8.2	9.4
25	.0179	1.92	2.52	3.0	3.6	4.3	5.2	6.1	7.1	8.0
26	.0159	1.67	2.14	2.60	3.2	3.8	4.5	5.3	6.1	6.9
27	.0142	1.44	1.84	2.25	2.73	3.3	3.9	4.6	5.3	6.0
28	.0126	1.24	1.61	1.95	2.38	2.85	3.4	3.9	4.5	5.1
29	.0113	1.08	1.41	1.73	2.10	2.51	2.95	3.4	3.9	4.4
30	.0100	.92	1.19	1.47	1.78	2.14	2.52	2.9	3.3	3.7
31	.0089	.77	1.03	1.28	1.54	1.84	2.17	2.52	2.85	3.2
32	.0080	.68	.90	1.13	1.36	1.62	1.89	2.18	2.46	2.76
33	.0071	.59	.79	.97	1.17	1.40	1.62	1.86	2.12	2.35
34	.0063	.50	.68	.83	1.00	1.20	1.41	1.60	1.80	1.99
35	.0056	.43	.57	.72	.87	1.03	1.21	1.38	1.54	1.71
36	.0050	.38	.52	.63	.77	.89	1.04	1.19	1.33	1.48
37	.0045	.35	.46	.57	.68	.78	.90	1.03	1.16	1.29
38	.0040	.30	.41	.50	.59	.68	.78	.88	.98	1.09
39	.0035	.27	.36	.42	.49	.58	.66	.75	.84	.92
40	.0031	.24	.31	.36	.43	.50	.57	.64	.72	.79

and C straight wire. These values apply to Chromel A but may be used for Chromel C with a discrepancy of less than 5 %.

Tables V and VI show the gage of the wire and the diameter of the arbor necessary to produce a coiled unit of a specified wattage. Again these data are meant only as a guide. They give

TABLE V. Coiled Units — 115 volts a.c.-d.c.

*CHROMEL-A • COILED UNITS • Watts—Gage—Arbor—Close Wound Length**

115 VOLTS

A.W.G. or B & S	LINEAL FEET	1500 WATTS		
		ARBOR DIAMETER		
		1/4	7/32	3/16
		Inches of Close-Wound Coil		
14	51.8	39.6	43.7	49.0
15	41.2	28.4	31.7	35.6
16	32.9	21.1	23.4	25.5

		1300 WATTS		
		ARBOR DIAMETER		
		1/4	7/32	3/16
15	47.5	32.8	36.6	41.0
16	38.0	24.4	27.0	29.5
17	29.6	16.8	18.7	20.7

		1100 WATTS		
		ARBOR DIAMETER		
		1/4	7/32	3/16
16	44.9	28.8	31.8	34.8
17	35.0	19.9	22.1	24.5
18	27.7	13.8	15.7	17.8

		900 WATTS		
		ARBOR DIAMETER		
		7/32	3/16	5/32
17	42.8	27.1	29.9	35.1
18	33.8	19.2	21.6	25.0
19	27.3	13.9	15.9	19.4

		700 WATTS		
		ARBOR DIAMETER		
		3/16	5/32	1/8
19	35.2	20.4	25.0	29.1
20	27.8	14.6	17.0	20.9
21	22.1	10.5	12.3	15.1

		500 WATTS		
		ARBOR DIAMETER		
		5/32	1/8	3/32
21	30.9	17.3	21.2	26.8
22	24.3	12.0	15.0	19.1
23	19.4	8.6	10.9	13.9

		300 WATTS		
		ARBOR DIAMETER		
		1/8	3/32	1/16
25	20.3	9.2	11.8	16.5
26	16.0	6.5	8.4	11.8
27	12.8	4.6	6.0	8.6

A.W.G. or B. & S.	LINEAL FEET	1400 WATTS		
		ARBOR DIAMETER		
		1/4	7/32	3/16
		Inches of Close-Wound Coil		
15	44.1	30.4	33.9	38.0
16	35.3	22.6	25.1	27.4
17	27.5	15.6	17.4	19.3

		1200 WATTS		
		ARBOR DIAMETER		
		1/4	7/32	3/16
16	41.2	26.6	29.2	31.9
17	32.1	18.3	20.3	22.5
18	25.4	12.7	14.4	16.3

		1000 WATTS		
		ARBOR DIAMETER		
		7/32	3/16	5/32
17	38.5	24.4	26.9	31.6
18	30.4	17.3	19.5	22.5
19	24.6	12.6	14.3	17.4

		800 WATTS		
		ARBOR DIAMETER		
		7/32	3/16	5/32
18	38.1	21.6	24.4	28.2
19	30.8	15.7	17.9	21.8
20	24.3	11.1	12.7	14.8

		600 WATTS		
		ARBOR DIAMETER		
		3/16	5/32	1/8
20	32.4	17.0	19.8	24.4
21	25.8	12.2	14.4	17.7
22	20.3	8.6	10.0	12.5

		400 WATTS		
		ARBOR DIAMETER		
		5/32	1/8	3/32
23	24.3	10.8	13.6	17.3
24	19.2	7.7	9.6	12.4
25	15.2	5.5	6.8	8.8

		200 WATTS		
		ARBOR DIAMETER		
		1/8	3/32	1/16
27	19.1	6.9	8.9	12.9
28	15.1	4.8	6.3	9.1
29	12.1	3.5	4.6	6.6

		100 WATTS		
		ARBOR DIAMETER		
		3/32	1/16	1/32
29	24.3	9.2	13.3	25.6
30	19.0	6.4	9.5	18.6
31	15.1	4.6	6.7	14.9

a concept of what the coil may be made from and what it will look like based on three different wire gages and three different arbor sizes.

TABLE VI. Coiled Units — 230 volts a.c.-d.c.

***CHROMEL-A** • COILED UNITS • Watts—Gage—Arbor—Close Wound Length**

230 VOLTS

A.W.G. or B. & S.	LINEAL FEET	3000 WATTS			A.W.G. or B. & S.	LINEAL FEET	2500 WATTS		
		ARBOR DIAMETER					ARBOR DIAMETER		
		1/4	7/32	3/16			1/4	7/32	3/16
		Inches of Close-Wound Coil					Inches of Close-Wound Coil		
14	104	79.3	87.3	98.0	15	99.0	68.3	76.2	85.3
15	82.5	56.8	63.4	71.0	16	79.2	50.7	51.2	61.3
16	65	42.3	46.9	51.2	17	61.7	35.0	39.0	43.2
		2000 WATTS					1800 WATTS		
		ARBOR DIAMETER					ARBOR DIAMETER		
		1/4	7/32	3/16			1/4	7/32	3/16
16	98.8	63.3	70.1	76.6	17	85.7	48.8	54.2	60.0
17	76.9	43.7	48.6	53.8	18	67.8	33.9	38.6	43.5
18	60.8	30.4	34.5	39.0	19	54.8	24.7	28.0	31.9
		1600 WATTS					1400 WATTS		
		ARBOR DIAMETER					ARBOR DIAMETER		
		7/32	3/16	5/32			3/16	5/32	1/8
18	76.1	43.3	48.8	56.4	19	70.4	40.9	50.0	58.2
19	61.6	31.4	35.8	43.7	20	55.6	29.1	33.9	41.8
20	48.7	22.2	25.5	29.7	21	44.2	21.0	24.6	30.2
		1200 WATTS					1000 WATTS		
		ARBOR DIAMETER					ARBOR DIAMETER		
		3/16	5/32	1/8			5/32	1/8	3/32
20	65.9	34.5	40.2	49.5	21	61.8	34.6	42.3	53.8
21	51.5	24.4	28.8	35.3	22	48.6	24.0	30.0	38.3
22	36.9	15.6	18.2	22.8	23	38.8	17.3	21.8	27.7
		900 WATTS					800 WATTS		
		ARBOR DIAMETER					ARBOR DIAMETER		
		5/32	1/8	3/32			5/32	1/8	3/32
22	54.1	26.7	33.4	42.5	23	48.6	21.6	27.3	34.7
23	43.3	19.3	24.3	31.0	24	38.4	15.4	19.2	24.8
24	34.2	13.7	17.1	22.1	25	48.6	11.1	13.7	17.7
		700 WATTS					600 WATTS		
		ARBOR DIAMETER					ARBOR DIAMETER		
		1/8	3/32	1/16			1/8	3/32	1/16
24	43.9	21.9	28.3	40.0	25	40.6	18.3	23.6	33.0
25	34.8	15.7	20.2	28.3	26	32.1	12.9	16.8	23.8
26	27.5	11.1	14.4	20.4	27	25.5	9.3	12.0	17.2
		500 WATTS					400 WATTS		
		ARBOR DIAMETER					ARBOR DIAMETER		
		1/8	3/32	1/16			1/8	3/32	1/16
26	38.5	15.5	20.2	27.6	27	38.4	14.0	18.0	26.0
27	30.6	11.1	14.4	20.7	28	30.6	9.8	12.8	18.5
28	24.2	7.8	10.2	14.7	29	24.4	7.1	9.3	13.3
		300 WATTS					200 WATTS		
		ARBOR DIAMETER					ARBOR DIAMETER		
		3/16	1/8	1/32			3/32	1/8	1/32
28	40.7	17.1	24.6	50.0	29	48.5	18.4	26.6	51.1
29	32.4	12.3	17.7	34.1	30	38.0	12.9	19.0	37.3
30	25.4	8.6	12.7	25.0	31	30.1	9.1	13.4	26.9

In all these coils the ratio of the I.D. of the coil to the diameter of the wire is $^4/_1$ to $^6/_1$. The O.D. of the coil will be approximately equal to the arbor diameter plus two times the wire diameter plus 0.005 in.

TABLE VII. Current-Carrying Capacity of Helical Coils

CURRENT CARRYING CAPACITY OF

CHROMEL-A and C HELICAL COILS

Approximate Amperes to heat Oxidized, Helical-Coiled wire in quiet air to given temperatures:
(Coil stretched to double the close wound length)

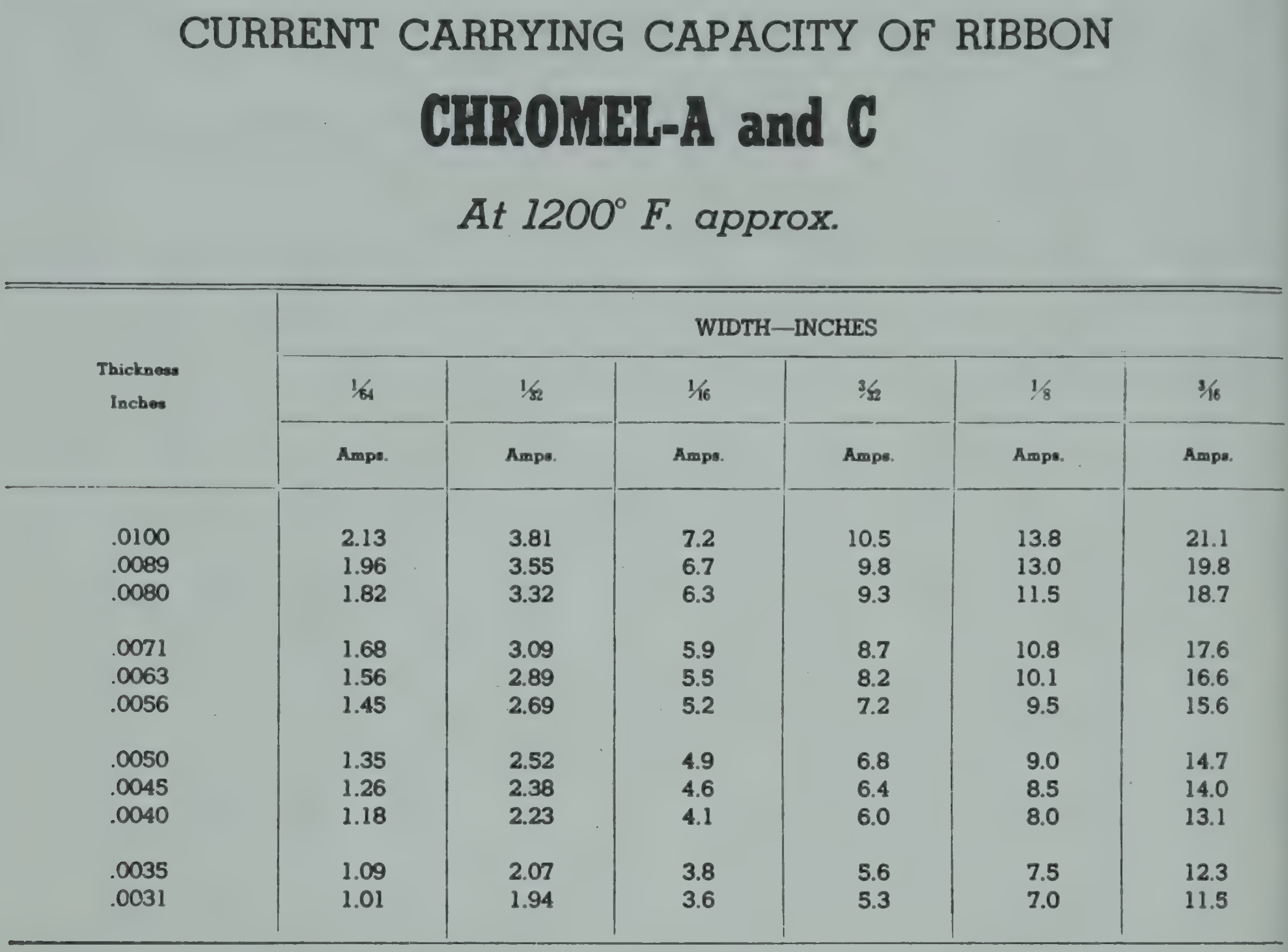
Degrees Fahr.			400	600	800	1000	1200	1400	1600	1800	2000
Degrees Cent.			205	315	427	538	649	760	871	982	1093
A. W. G. or B. & S.	Inches Wire Dia.	Arbor Dia. or Appr. I.D. of Coil	Amperes								
11	.091	3/16	7.9	12.9	18.0	23.4	29.2	35.7	42.9	50.9	59.7
12	.081	3/16	6.8	11.1	15.5	20.0	24.8	30.0	36.0	42.0	48.0
13	.072	3/16	5.8	9.7	13.5	17.3	21.3	26.0	31.0	35.5	41.0
14	.064	3/16	5.0	8.2	11.3	14.7	18.2	22.0	26.0	30.3	35.0
15	.057	3/16	4.3	7.0	9.6	12.3	15.2	18.5	22.0	25.3	30.0
16	.051	3/16	3.7	5.9	8.1	10.3	12.6	15.5	18.5	21.6	25.0
17	.045	3/16	3.1	4.9	6.6	8.5	10.6	13.0	15.5	18.0	21.0
18	.040	1/8	2.5	4.0	5.5	7.0	8.7	10.5	12.5	14.5	17.0
19	.036	1/8	2.05	3.3	4.6	5.9	7.3	8.9	10.5	12.0	14.0
20	.032	1/8	1.70	2.7	3.8	5.0	6.2	7.5	8.8	10.3	12.0
21	.0285	1/8	1.44	2.30	3.2	4.2	5.2	6.3	7.5	9.0	10.2
22	.0253	1/8	1.21	1.90	2.7	3.6	4.3	5.4	6.4	7.7	8.8
23	.0226	1/8	1.03	1.67	2.37	3.1	3.8	4.6	5.5	6.5	7.7
24	.0201	1/8	.89	1.44	2.07	2.7	3.4	4.1	4.9	5.7	6.7
25	.0179	1/8	.75	1.22	1.78	2.35	3.0	3.6	4.3	5.0	5.8
26	.0159	1/8	.63	1.04	1.49	2.00	2.6	3.1	3.7	4.4	5.0
27	.0142	1/8	.51	.86	1.22	1.65	2.2	2.65	3.1	3.8	4.3
28	.0126	1/8	.39	.68	.97	1.30	1.70	2.15	2.6	3.2	3.7
29	.0113	1/8	.30	.54	.78	1.03	1.35	1.75	2.1	2.6	3.1
30	.0100	1/8	.24	.42	.64	.86	1.13	1.42	1.75	2.1	2.5
31	.0089	1/16	.20	.35	.52	.70	.93	1.15	1.45	1.75	2.0
32	.0080	1/16	.164	.29	.43	.60	.78	.98	1.20	1.45	1.73
33	.0071	1/16	.133	.24	.35	.50	.66	.83	1.02	1.20	1.48
34	.0063	1/16	.110	.195	.28	.41	.54	.68	.84	1.00	1.20
35	.0056	1/16	.085	.155	.23	.33	.43	.54	.67	.81	.92
36	.0050	1/32	.070	.130	.196	.27	.35	.44	.54	.64	.75
37	.0045	1/32	.061	.112	.169	.23	.30	.38	.45	.54	.63
38	.0040	1/32	.052	.095	.143	.195	.25	.32	.38	.45	.53
39	.0035	1/32	.043	.079	.119	.164	.21	.26	.32	.37	.44
40	.0031	1/32	.035	.065	.096	.135	.170	.21	.26	.30	.36

To apply the figures from Tables V and VI, 7 % should be deducted from the length shown.

Table VII lists the current-carrying capacity of Chromel A and C helical coils. The values shown are for Chromel A but may be used for Chromel C with a discrepancy of not over 5 %.

In Table VIII the current-carrying capacity of Chromel A and C ribbon is listed. The current values in this table were based

TABLE VIII. Current-Carrying Capacity of Ribbon



CURRENT CARRYING CAPACITY OF RIBBON

CHROMEL-A and C

At 1200° F. approx.

Thickness Inches	WIDTH—INCHES					
	1/64	1/32	1/16	3/32	1/8	3/16
	Amps.	Amps.	Amps.	Amps.	Amps.	Amps.
.0100	2.13	3.81	7.2	10.5	13.8	21.1
.0089	1.96	3.55	6.7	9.8	13.0	19.8
.0080	1.82	3.32	6.3	9.3	11.5	18.7
.0071	1.68	3.09	5.9	8.7	10.8	17.6
.0063	1.56	2.89	5.5	8.2	10.1	16.6
.0056	1.45	2.69	5.2	7.2	9.5	15.6
.0050	1.35	2.52	4.9	6.8	9.0	14.7
.0045	1.26	2.38	4.6	6.4	8.5	14.0
.0040	1.18	2.23	4.1	6.0	8.0	13.1
.0035	1.09	2.07	3.8	5.6	7.5	12.3
.0031	1.01	1.94	3.6	5.3	7.0	11.5

on actual tests of single strands of oxidized ribbon, mounted to protect it from drafts and heated to 1200° C. In using this table it should be noted that Chromel C will operate at a slightly higher temperature than Chromel A.

Winding Coils

The winding of Chromel wire helical coil heating elements will be easier and more successful if the following rules are observed.

Ratio. The ratio of a coil is the relationship between its inside diameter and the diameter of the wire. The size of this ratio is important in determining the ease or difficulty of making a coil. A coil having a ratio of 2/1 or less is very difficult to make. If the ratio is 15/1 or more, the coil will be very flimsy and hard to handle and will also present "uneven stretch" problems.

De-Spooling. In making coils, the de-spooling operation is the source of much of the trouble. Overcoming the initial inertia

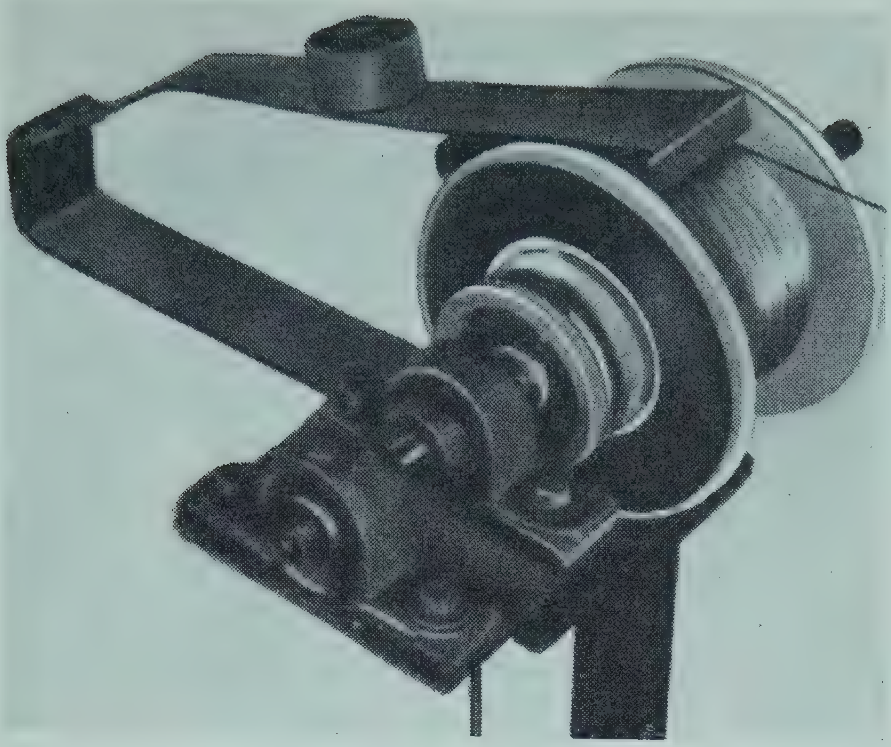
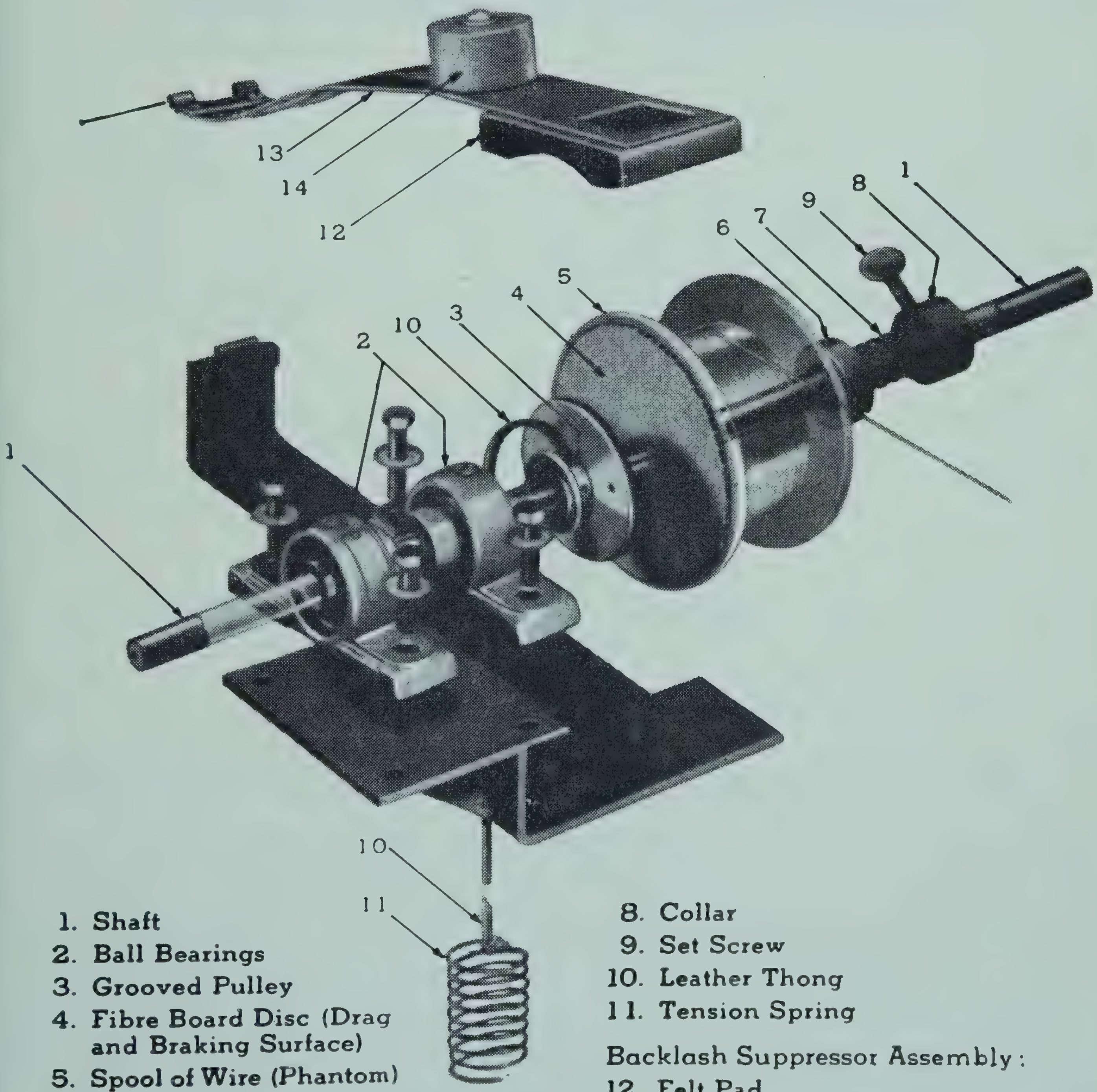


Figure 1A. De-spooler.



1. Shaft
2. Ball Bearings
3. Grooved Pulley
4. Fibre Board Disc (Drag and Braking Surface)
5. Spool of Wire (Phantom)
6. Thrust Bearing
7. Spring
8. Collar
9. Set Screw
10. Leather Thong
11. Tension Spring

Backlash Suppressor Assembly:

12. Felt Pad
13. Hinged Plate
14. Adjustable Weight

Figure 1B. Exploded view of de-spooler.

of the spool of wire, and then bringing it to a stop, are operations which produce tension in the wire that cause work-hardening. Both of these steps must be performed smoothly, without jerks, regardless of the speed at which the arbor is turning. Often these requisites are not met, in which case spot-hardening and uneven stretch result. If the wire is jerked too hard or run over pulleys that are too small, it may harden to a strong degree and result in a poor stretch. Figure 1A shows a de-spooling mechanism, or "take off" stand developed by the Hoskins Manufacturing Co. for use in making coils. Figure 1B is an exploded view of the de-spooler, showing the details of assembly and illustrating the principles of operation which meet the mechanical requirements for producing uniform coils.

Loading. The loading of a coil may be described as the tendency of each turn to crowd up against its neighbor. For best results a coil should be very slightly loaded and, above all, it must be uniformly loaded. Uniform loading is difficult because of variables created by the forward movement of the operator's hand. As an aid to uniform loading, the Hoskins Company has developed the coiling tool shown in Figure 2 (exploded view).

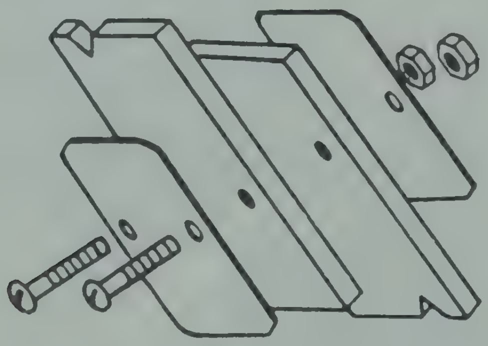


Figure 2. Exploded view of coiling tool.

This hand coiling tool gives good control over the feed angle of the wire as it goes onto the arbor. This is the operation in which loading is created and which must be uniform throughout the coil. The hand tool does not scratch the wire, and the wire does not touch the operator's hand. The coiling tool may be used mechanically as well as by hand. When long coils or coils of bad ratios are to be made, the coiling tool is mounted as shown in Figure 3.

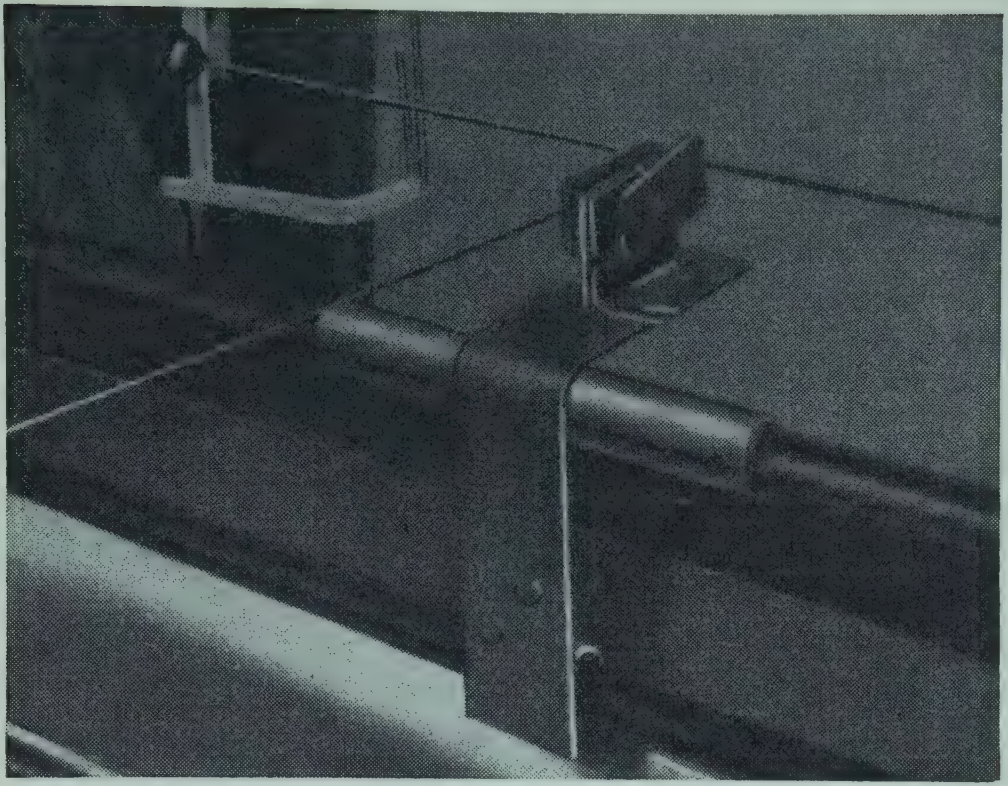


Figure 3. Coiling tool mounted on lathe.

Useful Life Expectancy of Chromel A

The Hoskins Manufacturing Co., in cooperation with the American Society for Testing Materials, developed a method of testing heating element material. The chart shown in Figure 4 gives the typical life curve of two metal-chromium alloy wires A and C, respectively. Chromel A has the very desirable characteristic of nearly constant hot resistance, which means that a heating element made from Chromel A wire will deliver full rated power throughout its entire life. Chromel C, on the other hand,

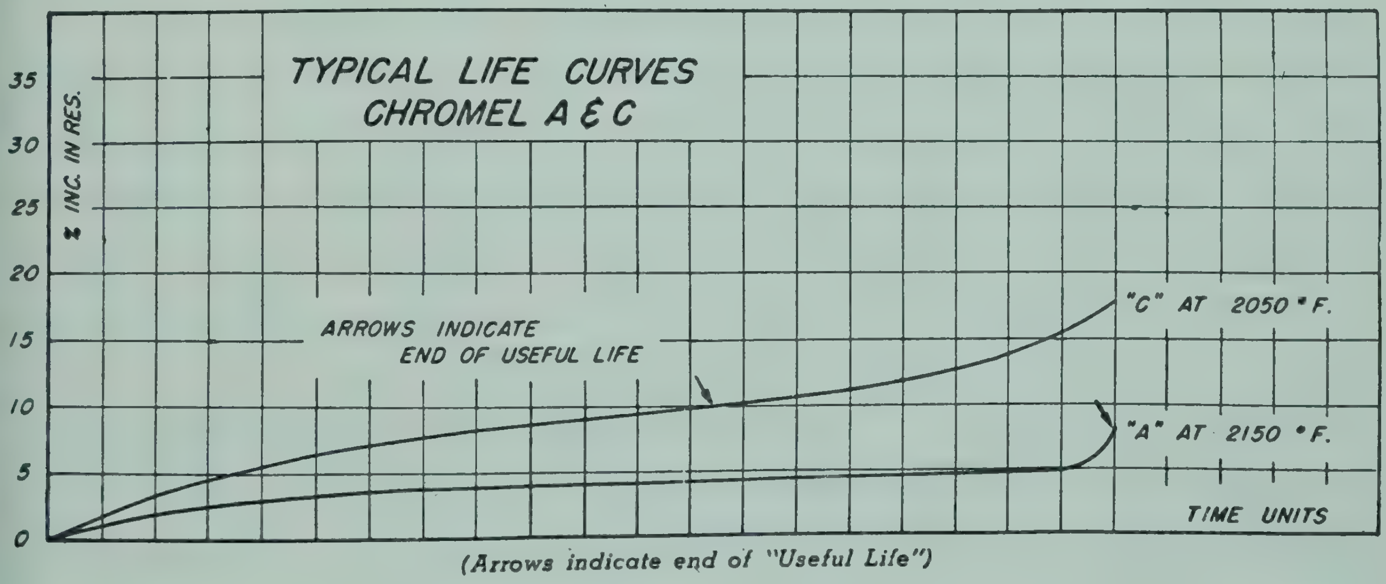


Figure 4. Curve showing life expectancy of Chromel wire.

shows a steady increase in hot resistance. This increase and accompanying decrease in heating ability tend to prolong the total life of the wire. Therefore, the useful life (time to reach 10 % increase) is a sounder basis for judging these two wires.

Single- and Three-Phase Heating Units. When three heating units are to be operated from a three-phase circuit, they are connected either in "Star" or "Delta". In either of these connections, the total power consumed is: (voltage between line wires) times (current in each line wire) times (1.73) or by formula: watts = volts × amperes × 1.73. For the Delta connection, the three elements are all connected in series in the form of a triangle; one line wire is connected to each corner of the triangle, and each unit draws one-third of the total power. For example, if the total power is to be 3 kilowatts, and the line is 220 volts three-phase, then each unit is designed to draw 1 kilowatt at 220 volts.

For Star connections, each of the three elements is again designed for one-third of the total power. Then, one end of each unit is connected at a common point called "neutral point." The three other ends are connected to the three line wires. In this case, the individual units are designed not for line voltage but for voltage between the line and the neutral point which, in turn, is equal to the line voltage divided by 1.73. For example: if the three units combined are to draw 3 kilowatts and are connected in Star to a 220-volt, three-phase line, then each unit is designed to draw 1 kilowatt at 220/1.73 or 127 volts.

Fundamentally then, single- and three-phase units are designed in the same manner, but the single-phase unit and each of the three elements of a three-phase Delta unit are designed for line voltage. For a three-phase Star unit, each of the three elements is designed for the voltage between the line and the neutral point, or line voltage divided by 1.73.

XVI

MISCELLANEOUS EQUIPMENT AND PROCEDURES

Apparatus for a few of the standard chemical processes which were not covered in the preceding chapters are described in this chapter. This does not imply that these techniques are less important than those previously described, and this is especially true in reference to chromatography. Chromatography is a relatively new field and one in which new equipment and procedures are constantly being reported.

CHROMATOGRAPHY

Chromatography has become so widely used in the past few years that any glassblower should have some knowledge of what can be accomplished by chromatography and what type of equipment is needed. In essence chromatography is the process of separating chemical compounds by the partition of a solute between two solvent systems, usually liquids one of which is moving in respect to the other. There are three types of chromatography, partition, adsorption, and ion-exchange.

Partition Chromatography

This process makes use of the principle of separating one solvent from another by shaking the solution with a third solvent in which one of the solutes is more soluble than the other. Any desired degree of separation may be obtained by repeating the shaking process with fresh solvent. In contrast to this slow tedious step-by-step process of separation, partition chromatography is the process of applying mixed solutes to the top of a column of solid substance which holds one liquid phase static while a second phase is allowed to flow continuously down the column. Martin and Synge[1] introduced the use of silica gel wetted with water as the fixed phase and chloroform as the moving phase. Although it is reported that Shonbien was the first to separate substances by adsorption, Consden, Gordon, Martin, and Synge[2] are credited with the introduction of paper chromatography, in which paper is substituted for silica gel as the solid holding substance. They reported that a small amount of water held by adsorption on paper when in equilibrium with moist air was sufficient to permit the chromatography process to work. The same authors stated that the efficiency of contact between the liquid phases is greater in chromatography than in ordinary distillation and extraction columns.

Adsorption Chromatography

Adsorption chromatography depends on a column of finely divided adsorbent wetted by the one liquid moving phase. The material to be separated is dissolved in a suitable solvent and applied to the top of the column. The amount of material applied to the top of the column is kept small in comparison to the total adsorption capacity of the column, and when the column is sufficiently loaded with material, a pure solvent is allowed to run through the column. In this respect adsorption chromatography is similar to partition chromatography, since in both cases the solutes gradually separate as they are drawn down the column at different rates by the flowing solvent.

Ion-Exchange or Displacement Chromatography

In this method the column is filled with an ion-exchange resin, some types of which are commercially available now. These commercial resins are chemically stable, have a high adsorption capacity, and are monofunctional, that is, each resin has only one type of chemical group which can take part in the ion-exchange reaction. The method of operation for ion-exchange chromatography is similar to that for partition chromatography in that a limited amount of the liquid to be resolved is applied to the top of the column; however, instead of flushing the column with pure solvent, a developing solution is used. Since this solution contains a solute which is more strongly adsorbed *on* the column than any of the other solutes in the mixture, it displaces the original solute as it flows down the column. This developing solute gradually pushes the mixture of solutes farther and farther down the column in a series of bands, the least strongly adsorbed solute constituting the first band and so on until the most strongly adsorbed solute is displaced by the developing solute. Because of the overlap between one band and the next, ion-exchange chromatography is not suitable for quantitative separations; however, the advantage of this type of separation is that high concentrations of solute can be used.

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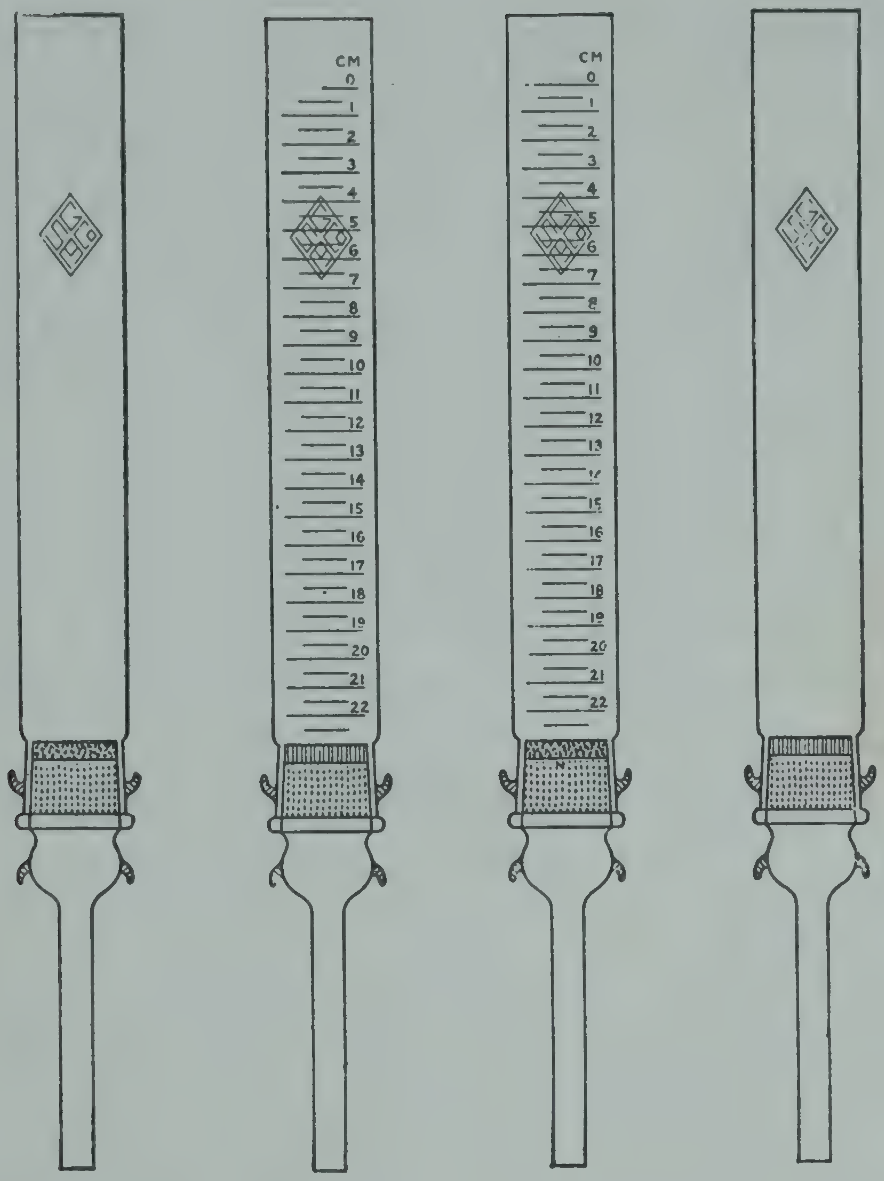
[1] Martin, A. J. P., and Synge, R. M. L., *Biochem J.* (*London*), **35**, 1358 (1941).

[2] Consden, R., Gordon, A., Martin, A., and Synge, R., *Biochem J.* (*London*), **38**, 224 (1944).

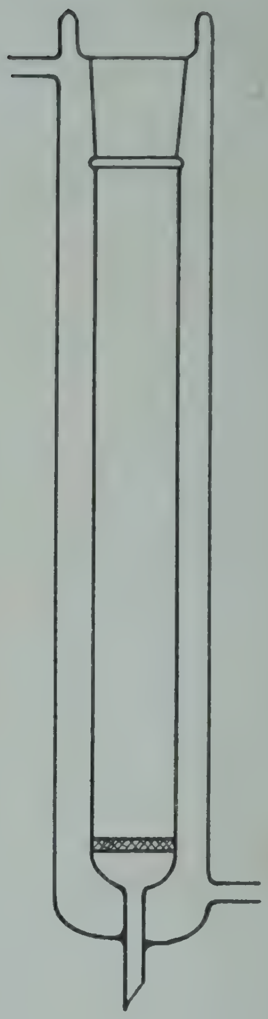
Chromatographic Equipment

Column Chromatography. Usually glass tubes are used for column chromatography, although quartz, metal, and plastics such as Lucite have been used. The size of chromatograph tubes varies from a few millimeters in diameter to several centimeters, and the working height from a few inches to several feet, depending on the amount and concentration of the material being worked with. The adsorbent is usually supported on some kind of a porous diaphragm, the most suitable being a fritted glass plate sealed into the column at the bottom. The tube should be sufficiently long so as to leave a space above the absorbent to contain a supply of liquid, either of the original solution or of the developer. A bulb may be sealed at the top of the column to form this liquid reservoir; however, if a bulb is used the neck of the bulb should be larger than the column to avoid difficulties in filling or emptying the column. Figures 1A and 1B show columns suitable for partition, adsorption, or ion-exchange chromatography.

For chromatograms which are run with the aid of gentle suction at the bottom of the column, the chromatographic tube shown in Figure 1C may be used. This same tube may be used for pressure chromatograms. In Figure 1D is shown a pressure head



A

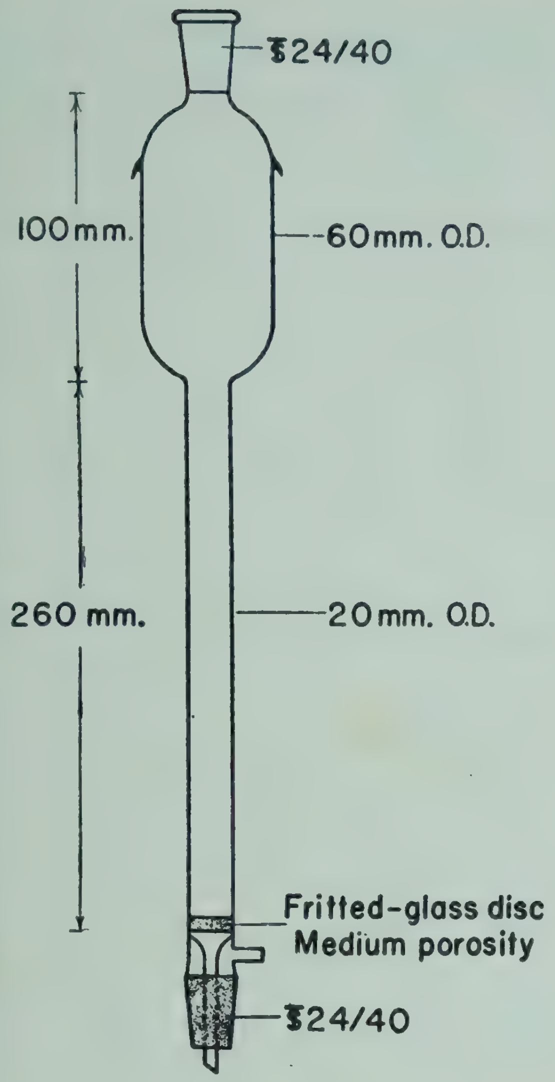


B

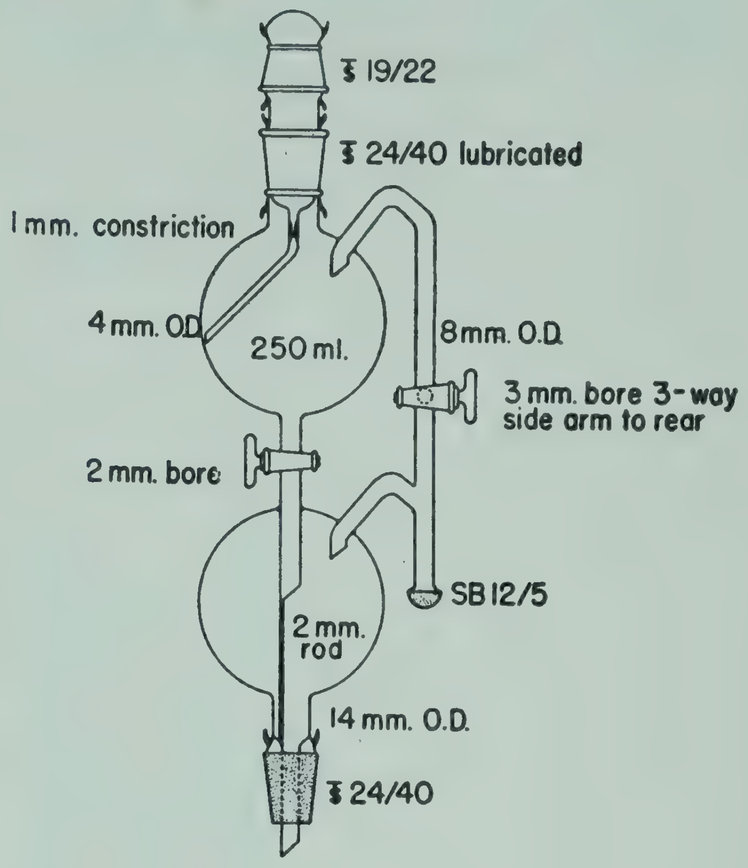
Figure 1A. Glass columns for column chromatography. (Courtesy of Scientific Glass Apparatus Co., Inc.)

Figure 1B. Jacketed chromatography column.

which may be used with the pressure column (Figure 1C). This head eliminates the difficulties commonly encountered in introducing the solution and solvents, namely, disturbance of the packed adsorbent when the pressure is released. The developing or eluting solutions are introduced through the 24/40 $ joint which should be thoroughly lubricated and is rotated as the solution is added to rinse the walls of the upper flask. When the solvent is in place in the upper flask the 19/22 $ cap is set on and the 3 mm. stopcock (side pressure manifold) opened to equalize the pressure in both bulbs. When the pressure has stabilized, the 2 mm. stopcock in line between the two bulbs is opened, and the solvent


Ŧ24/40
100 mm.
60 mm. O.D.
260 mm.
20 mm. O.D.
Fritted-glass disc
Medium porosity
Ŧ24/40

C


Ŧ 19/22
Ŧ 24/40 lubricated
1 mm. constriction
4 mm. O.D.
250 ml.
8 mm. O.D.
3 mm. bore 3-way side arm to rear
2 mm. bore
SB 12/5
2 mm. rod
14 mm. O.D.
Ŧ 24/40

D

Figure 1C. Column for pressure or suction chromatography. (From F. A. Gunther and R. C. Blinn, *Analysis of Insecticides and Acaricides*, Interscience, New York, 1955.)

Figure 1D. Pressure head for chromatography. (From F. A. Gunther and R. C. Blinn, *Analysis of Insecticides and Acaricides*.)

will then drain cleanly down the rod and into the chromatograph column without splashing.

In most cases pressure chromatography is carried out at from 4 to 6 p.s.i. It is important that the pressure regulator used in conjunction with pressure chromatography be both sensitive and accurate, and it is advisable to insert a 5-gallon surge tank between the regulator and the air inlet (SB 12/5). When several pressure columns are to be operated at one time, it is better to operate each one with a separate pressure regulator, rather than run them all from a manifold.

Paper Chromatography. In Figure 2 is illustrated the necessary apparatus for paper strip chromatography. A battery jar serves as a container and a piece of plate glass should be set on top of the jar to seal it off and maintain the atmosphere of the container saturated with the solvent used.

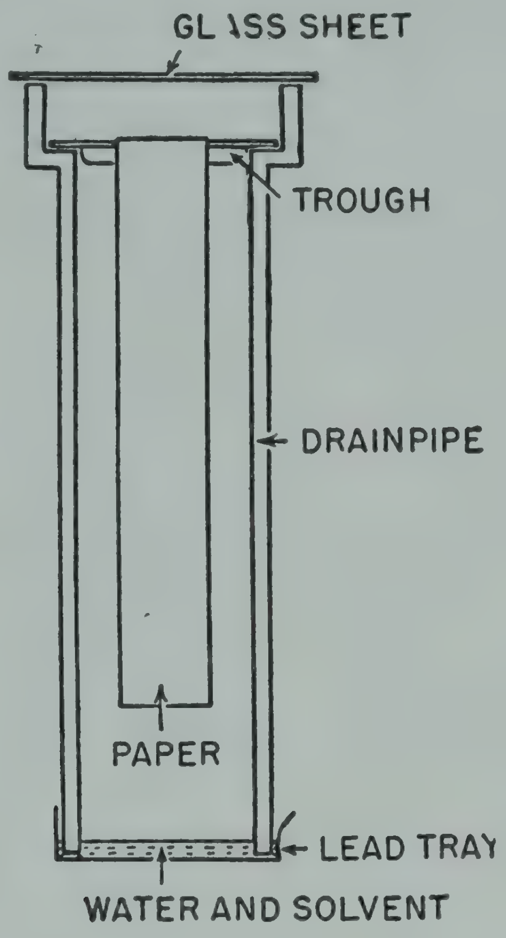


Figure 2. Apparatus for paper strip chromatography. (From A. Weissberger, *Technique of Organic Chemistry*, Vol. V.)

The apparatus for large paper chromatography is shown in Figure 3. The rack for supporting the troughs and paper should be made from 8 to 10 mm. solid glass rod, while the troughs may be made from 22 mm. O.D. tubing with the top half sawed or ground off.

In Figure 4 is seen a cross section of a chromatography trough with the ends of the paper immersed in the developing solvent. A glass rod bent up at the ends to form handles may be used to weight down the ends of the paper and hold it in the developer, while additional rods fused to the frame on each side of the trough hold the paper away from the edges of the trough and prevent siphoning. The spots of mixture should be placed just below these rods. Care should be observed in making and annealing the solid rod support frames to be sure they are free rom strains. When annealing is done in an electric oven the

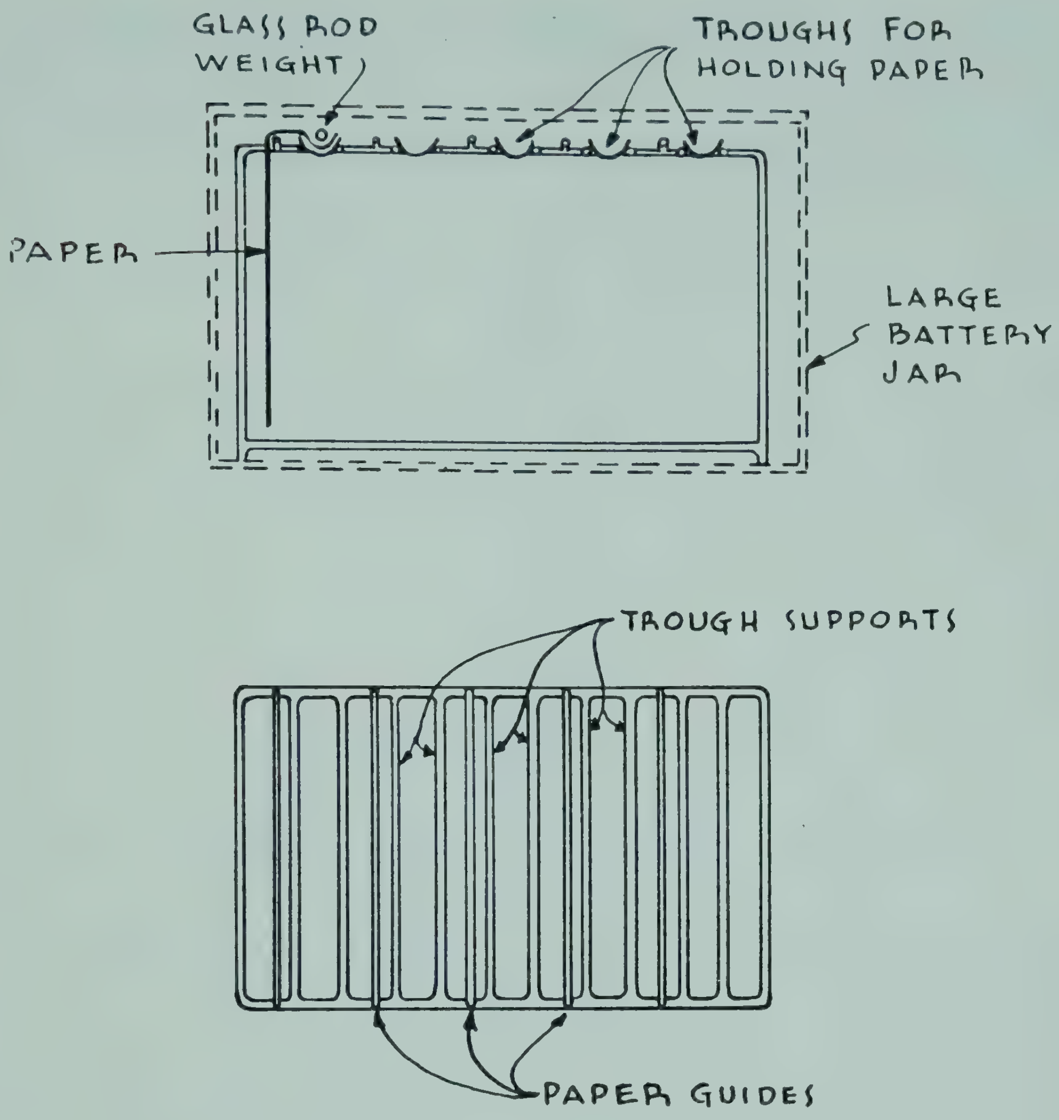

GLASS ROD WEIGHT
TROUGHS FOR HOLDING PAPER
PAPER
LARGE BATTERY JAR
TROUGH SUPPORTS
PAPER GUIDES

Figure 3. Apparatus for large paper chromatography.

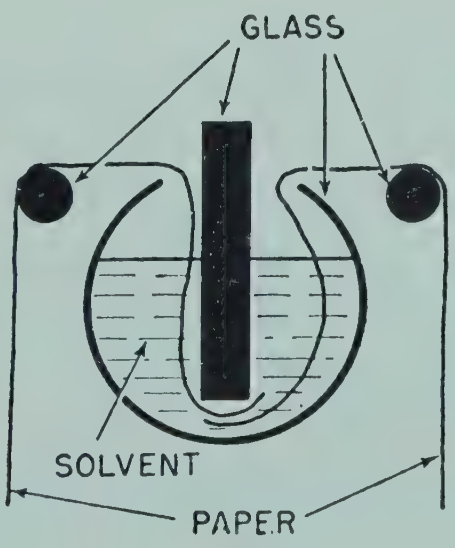

GLASS
SOLVENT
PAPER

Figure 4. Cross section of a chromatography trough. (From A. Weissberger *Technique of Organic Chemistry,* Vol. V.)

frames should be well supported to prevent sagging.

Since the apparatus previously shown in Figures 3 and 4 are rather fragile, Figure 5 shows a simple arrangement for making paper chromatograms. This can be made from a large test tube or measuring cylinder. In use the paper strip with the test spots is suspended from the rubber stopper by means of a bent paper clip. It is hung so that the lower end of the paper does not quite touch the surface of the solvent. After sufficient time is allowed for the paper to come into equilibrium with the water and solvent vapors, the wire hook is pushed further down into the rubber stopper, allowing the lower end of the paper end of the paper to dip into the solvent, thus starting the chromatographic process.

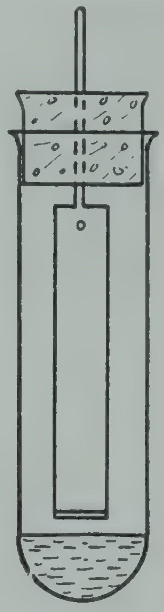


Figure 5. Micro paper chromatography.

CRYSTALLIZATION

Simple crystallization is used principally for the purification of a reaction product or similar material, made up largely of a single substance and having only a small percentage of impurities present. Crystallization consists essentially of the following steps.

1. The substance is dissolved in a solvent at the boiling point.

2. The hot solvent should then be filtered to remove any insoluble impurities.

3. The filtered solution is then allowed to cool and deposit crystals of the substance.

4. The crystals are then separated from the supernatant liquid (solvent).

5. The crystals should be washed to remove any adhering solvent.

6. The crystals should be dried to remove any last trace of solvent.

The selection of a suitable solvent for the crystallization is very important. A good solvent should dissolve a large quantity of the material at elevated temperatures, but only a small quantity at low temperatures. In practice it is desirable to use slightly more than the minimum quantity of hot solvent (2 % to 5 % more) required to dissolve the sample, so that the hot solution will not be quite saturated with solute. This aids in preventing separation of the crystals due to the slight cooling during filtration. In addition to the solvent's dissolving the material, it must readily dissolve the impurities, and it must be easily removed from the crystals of the purified substance. It is equally important also that the solvent not react with the material being precipitated.

The equipment necessary for simple crystallization consists of a suction filter flask, a Buchner funnel, and some filter paper. There are, however, many special methods and techniques of crystallization such as partial freezing, fractional freezing, slow freezing, and enclosed freezing. These are described in *Technique of Organic Chemistry, Volume III*,[3] with illustrations of the equipment needed for each method.

EXTRACTION WITH SOLVENTS

The process of extraction is used in chemistry for the separation and isolation of substances in solids, for the isolation of dissolved substances, and for the removal of soluble impurities from a mixture. Extraction is also used to remove the solute to an environment more suitable for study or isolation. For this latter purpose, extraction has long been one of the most widely used processes in the laboratory.

A few of the methods of extraction are discussed below.

[3] Interscience, New York, 2nd ed., Part I; 1956.

Discontinuous Extraction

In this method a given volume of solute is mixed with a given volume of solvent by being shaken in a separatory funnel. The funnel should be of such size that it is not more than three-fourths filled by the solute and solvent. After the funnel has been shaken a sufficient time for equilibrium to be established between the solute and solvent, it is allowed to stand undisturbed until the layers have separated competely. Vigorous shaking is desirable, provided it does not produce an emulsion that interferes with the subsequent separation.

Multiple Extraction

This method of bringing the solute and solvent together many times may be done the same way as the single extraction, except that several separatory funnels, tubes, flasks, or other containers are used, and the solute brought in contact with fresh solvent during each shaking.

Continuous Extraction

A common form of apparatus for continuous extractions involving solids is called a Soxhlet extractor and is shown in Figure 6. In this apparatus the solvent is boiled in the flask and passes through the side arm into the condenser, which directs the condensate onto the mass of solid held in a paper thimble. A small return line is so constructed that solvent remains in the extractor until the liquid level reaches the top of the return line, at which time a siphoning action begins which empties the extractor of solvent. The extraction process is a continual cycle of the slow filling of the extractor with solvent, of the siphoning out of the liquid, and the refilling of the extractor. A return line which will siphon properly is the only critical feature of an apparatus for the successful extraction from solids. In small extractors with return lines of 6 to 9 mm. O.D. this problem is not serious, but larger extractors with return lines of 10 to 12 mm. O.D. and over pose a problem. It is helpful if the extractor is designed to allow the liquid level to be several inches above the top of the return line.

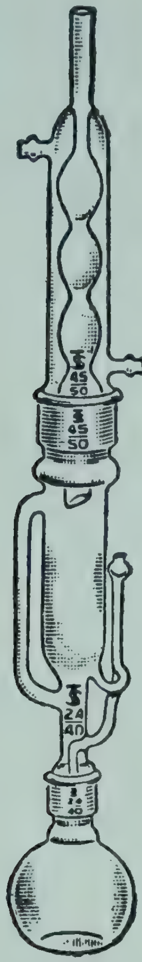


Figure 6. Soxhlet extractor. (From A. Weissberger, *Technique of Organic Chemistry,* Vol. III, 2nd ed., Part I.)

Liquid-Liquid Extractions

The extraction of a dissolved substance in a solution is accomplished by extraction with an immiscible solvent. The general principle underlying this process is known as the law of distribution, which states "In a dilute solution a substance distributes itself between immiscible solvents so that the ratio of concentration in the second solvent always remains constant (at constant temperatures)." Figure 7 shows an apparatus for liquid-liquid extractions using lighter solvents.. The solvent is boiled in a flask on the side arm, the vapor passing to the condenser, which directs the condensate into a funnel and so through a fritted plate bubbler (glass). There it is broken up into fine particles or bubbles before passing through the liquid from which the substance is being extracted. Liquid-liquid extractors of this design may be made of any size tubing desired, providing the condenser is made of proportional size. Fritted glass bubblers of several sizes may be obtained from the Corning Glass Works.

A liquid-liquid extractor for extractions with a heavier solvent is shown in Figure 8. In this extractor the solvent is boiled in the flask on the side arm, the vapor passes up to the condenser where it condenses and falls as drops of condensate.

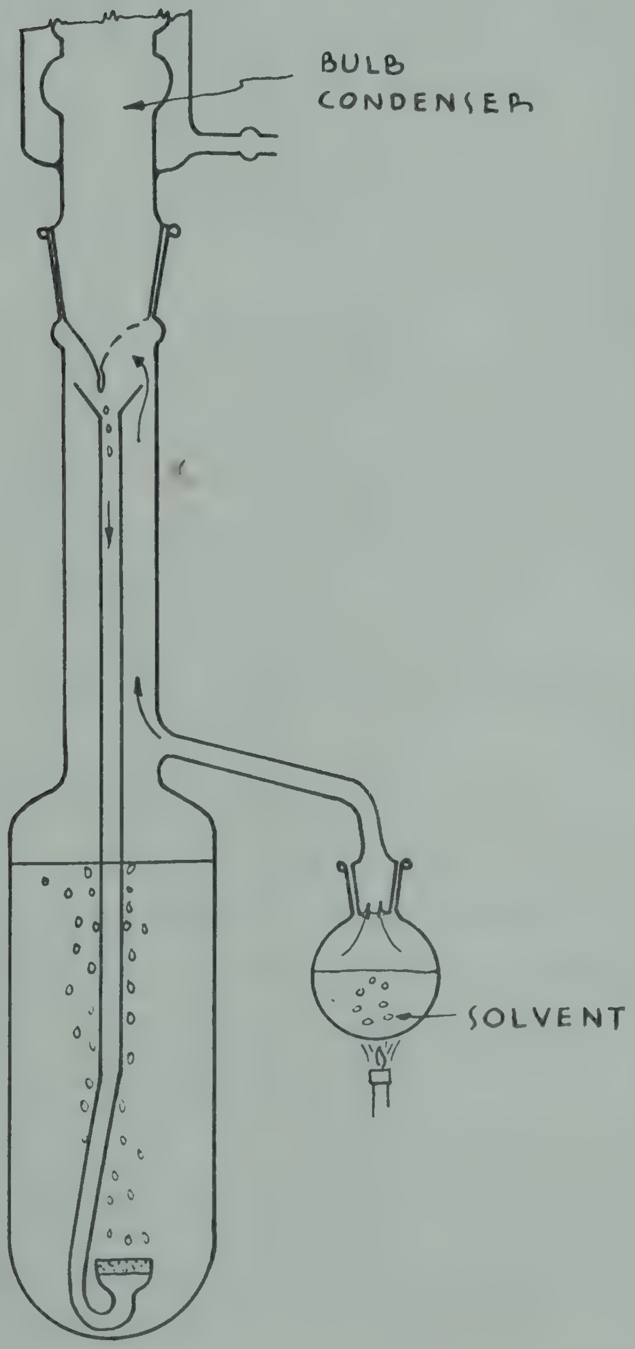


Figure 7. Liquid-liquid extractor for extraction with a lighter solvent.

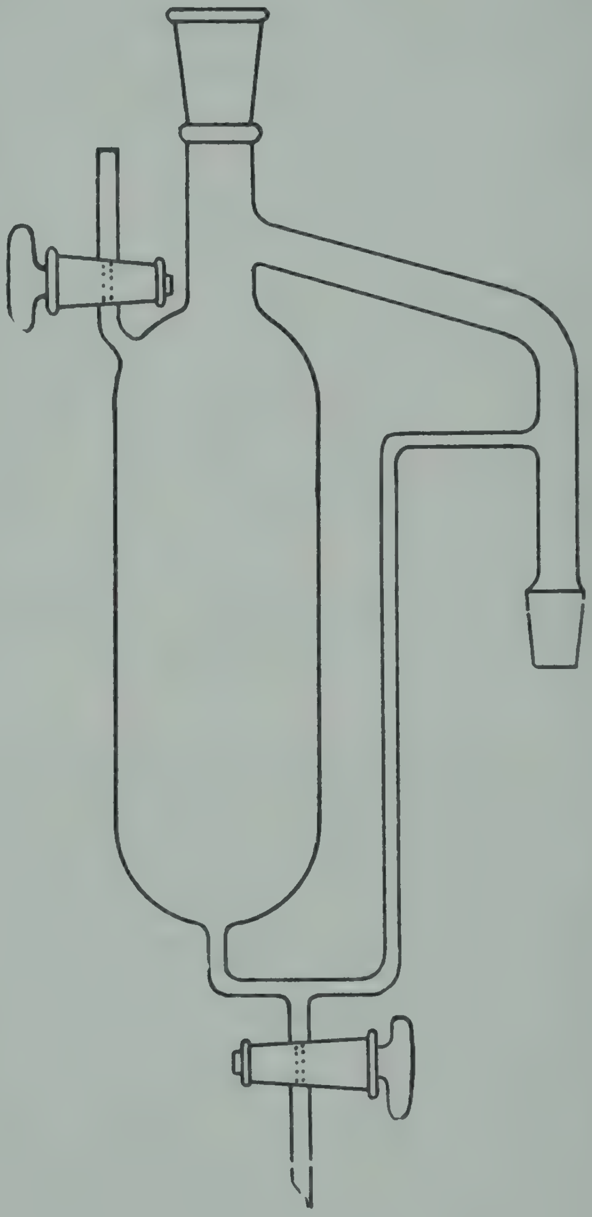


Figure 8. Liquid-liquid extractor for extraction with a heavier solvent.

The extraction process occurs as the drops of solvent pass down through the liquid to the bottom of the extractor. Gradually the solvent siphons back into the boiling flask and the cycle is repeated again and again until the extraction is completed.

FILTRATION

Filtration is one of the most common operations employed in practical organic chemistry. The use of filtration in chemical laboratories as early as A.D. 1515 has been disclosed in writings of that time. Also included in these writings were complete descriptions of some of the various types of filtration apparatus used.

Filtration has many objectives, such as:

1. The collection of the solid from a liquid. The product of a reaction may be obtained by filtering the precipitate from a liquid.

2. The removal of excess liquid from a solid. This is a preliminary step in the drying of many solids.

3. The purification of a solid. This purification is accomplished by dissolving the solid in a solvent in which it is sparingly soluble, but in which the impurities are more soluble. The desired purity may be obtained by repeating this operation as often as necessary.

4. The change in liquid phase. Solids may be changed from one liquid phase to another by filtering and then dissolving the solid in a second liquid phase.

5. The recovery of a liquid. This is a common objective of filtration and the procedure to be adopted depends on whether it is also desired that the solid be saved.

6. The removal of foreign matter such as dirt and fibers. In many preparations the only objectionable impurity is a small amount of foreign matter which may have come from the apparatus, the atmosphere, or accidental causes.

7. The removal of processing material. Liquids which have been treated with adsorbents, decolorizing agents, etc. must be freed of these solid materials before being used.

8. Analytical purposes. Many analytical procedures require extreme purity of solutions and precipitates. Filtering techniques and apparatus for qualitative and quantitative analytical purposes are highly developed, and the special techniques should be obtained from articles or books devoted to filtration.

The most common medium for laboratory filtration is filter paper in the form of circles. These are readily folded or fluted for use with filtering funnels. The funnel may be made of glass, rubber, metal, enamel, plastic, etc. Glass funnels come in a wide variety of sizes, with both long or short stems, and may be adapted to either hot or cold filtering. In Figure 9 are shown several designs of filtering sticks. Some of these filter sticks may be purchased commercially; others may be made by a glassblower.

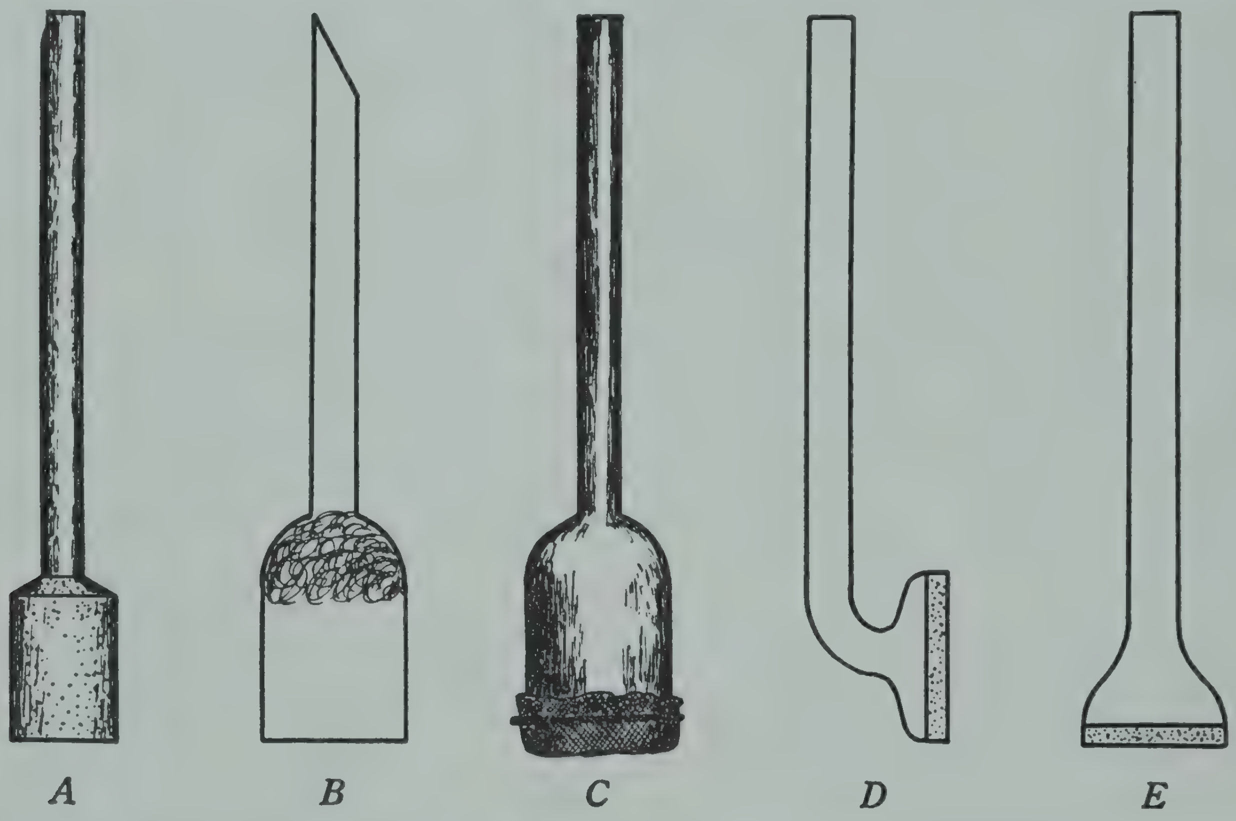


Figure 9. Filter Sticks. (From A. Weissberger, *Technique of Organic Chemistry*, Vol. III, 2nd ed., Part I.)

FREEZING AND MELTING POINT TEMPERATURES

The freezing and melting points of a pure compound are the temperatures at which solid crystals of the compound are in equilibrium with the liquid phase under its own vapor pressure. When this equilibrium condition is reached by cooling the liquid, the temperature is known as the freezing point; when it is reached by heating the compound, the temperature is known as the melting point.

An apparatus for determining the freezing point consists of a jacket which permits cooling the sample at a slow steady rate (unsilvered vacuum Dewars are ideal for this), an inner sample tube which may be heated to melt the sample, a stirrer to maintain the sample at equilibrium, and a thermometer or thermocouple to register the temperature of the sample. The apparatus designed by Moir, Glasgow, and Rossini[4] meet these specifications and are shown in Figure 10. Lynn[5] described a very precise apparatus for determining the freezing point of samples as small as a gram (Figure 11).

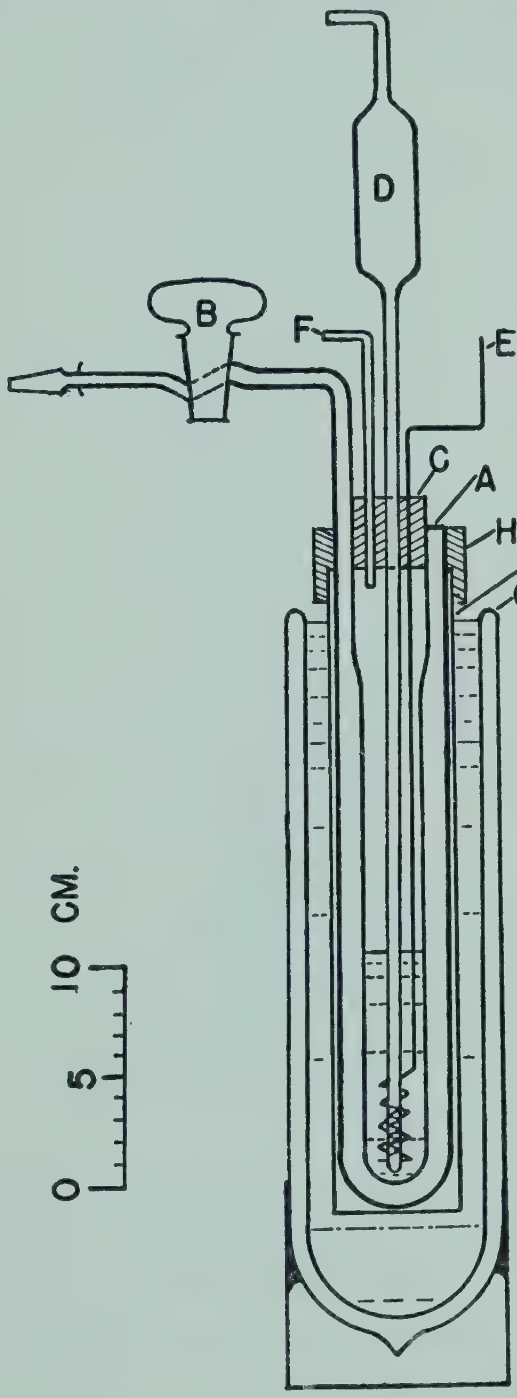


Figure 10. Freezing point apparatus. (From A. Weissberger, *Technique of Organic Chemistry*, Vol. I, 2nd ed., Part 1.)

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[4] Moir, H. F., Glasgow, A. R., and Rossini, F. D., *J. Research Natl. Bur. Standards*, **26**, 591 (1941).

[5] Lynn, G., *J. Phys. Chem.*, **31**, 1381 (1927).

Molecular weight determinations may be made by means of the freezing point. For complete information on this process the section entitled "Freezing point molecular weight determinations" in *Physical Methods of Organic Chemistry* Volume 1, Part 1,[6] should be consulted.

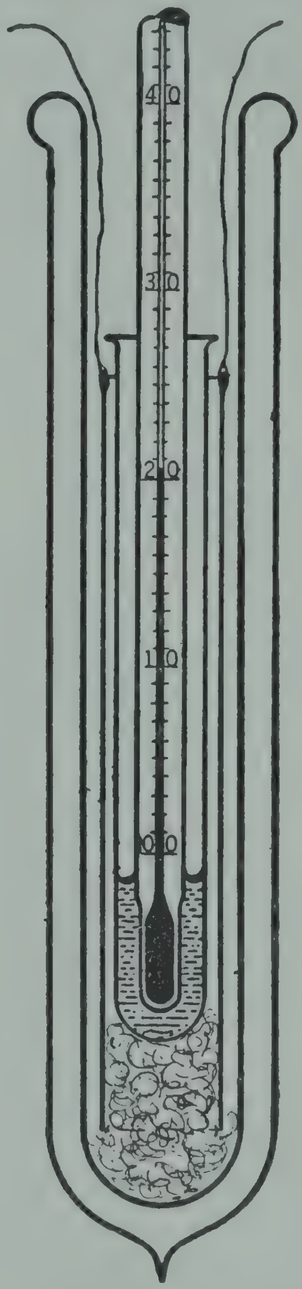

Figure 11. Micro freezing point apparatus. (From A. Weissberger, *Technique of Organic Chemistry*, Vol. I, 2nd ed., Part I.)

One of the most important applications of the melting point is its use as a method of determining purity. When a substance is pure the melting point is usually sharp, whereas with mixtures, melting is usually over a wide temperature range.

To determine the melting point of a crystalline substance, a small amount of the finely powdered substance is placed in a thin-walled capillary tube, which is fastened to or placed near the thermometer in a well-stirred and regulated heating bath of paraffin oil, glycerol, or silicone oil. The temperatures recorded are that at which the crystals commence to liquefy and that at which they are completely liquefied. The melting-point range (the temperature range between the beginning of liquefaction and complete liquefaction) and the actual temperature of melting indicate the purity of the substance.

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[6] Second edition, *Interscience*, New York, 1949.

A pure crystalline organic substance usually possesses a sharp melting point, that is, it melts completely over a short temperature range.

Two important factors to be considered in building a melting-point bath are the heating element and the stirrer.

1. The heater should be sufficiently sensitive that an even and accurate control of the rise in temperature is maintained during the taking of a melting point. A rise in temperature of about 2° C. per minute is satisfactory for ordinary work. Too fast a temperature rise does not allow for the necessary time lag required for the melting process to occur, resulting in an observed temperature range larger than the true one. Too fast a temperature rise will also cause the thermometer reading to be lower than the actual temperature of the bath because of the time required for heat transfer to the mercury. This will cause the observed temperature to differ from the actual temperature of melting.

2. Steady and thorough stirring of the liquid in the bath is necessary to prevent the liquid from circulating in hot and cool stria. Open-coil electric heaters sealed inside the apparatus and immersed in the liquid help create the turbulence necessary for efficient, even heating. Improper heating or stirring are indicated when the temperature does not remain constant, or when a different temperature may be obtained by moving or relocating the thermometer in the apparatus.

LYOPHILIZATION

This is the process of drying a material by subliming the ice. The lyophile apparatus shown in Figure 12 was designed by

Campbell and Pressman[7] and is satisfactory for small-scale laboratory work. The condensing surface of the inner cold finger is tapered to permit the accumulation of a greater amount of ice.

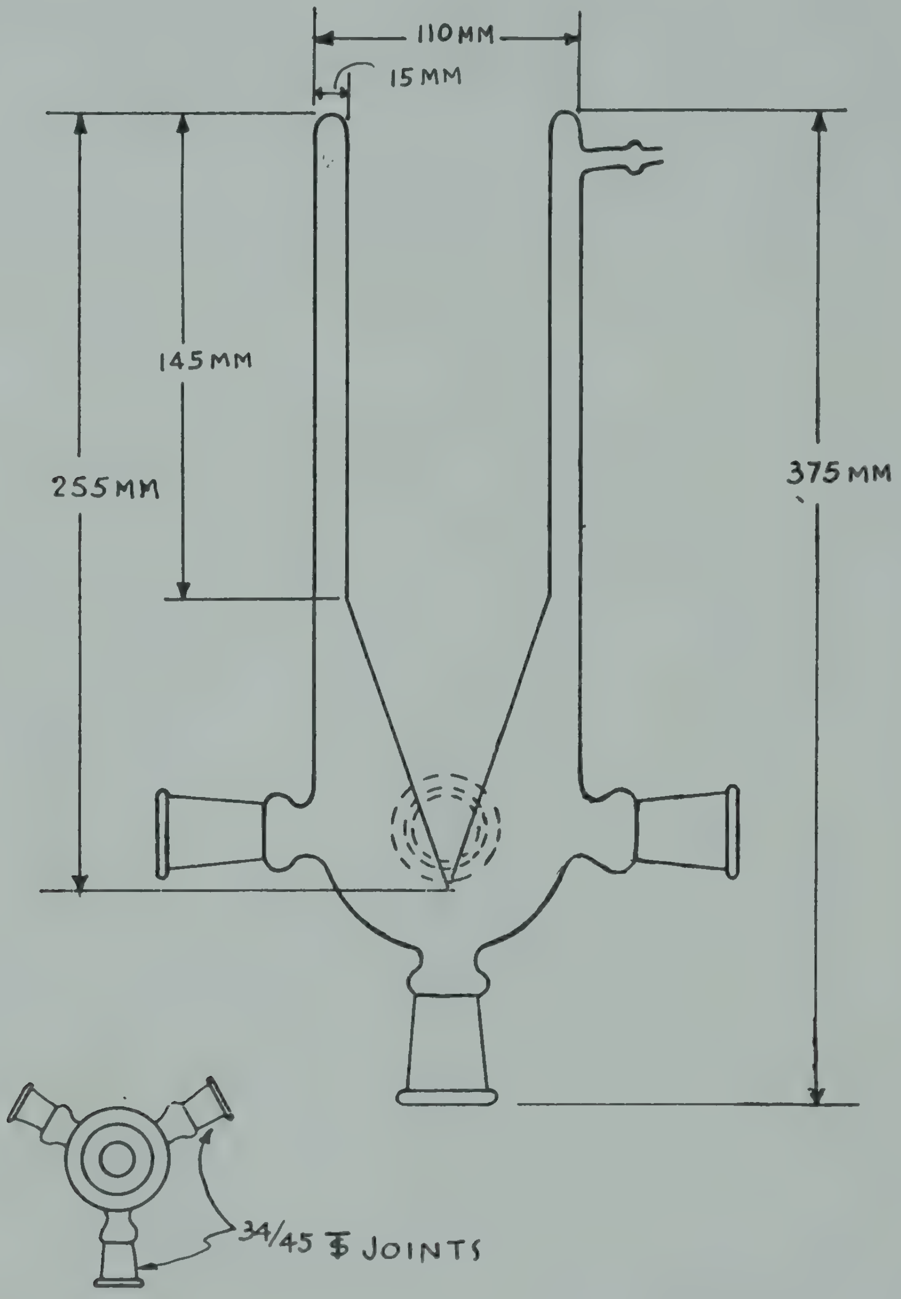

Figure 12. Lyophile apparatus.

A lyophilizer with the dimensions given will hold about 400 ml. of ice on the cold finger. Pear-shaped flasks should be used in order to facilitate removal of the dried material. The flasks are prepared for use on the lyophilizer by being filled to about 25 % of their capacity. They should then be held at an angle and rotated in a dry ice-acetone freezing mixture until all the material has frozen solidly to the side walls. This procedure will result in an even layer of material on the walls.

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[7] Campbell, D. H., and Pressman, D., *Science*, **99**, 285 (1944).

During operation the cold finger cone is filled with a mixture of dry ice and alcohol and, when all the flasks are prepared and attached, the assembly is evacuated. Unused $ openings to the condenser may be capped with sealed-off $ joints, which may also be used for drying small amounts of sample.

The temperature inside the flasks will depend on the rate of evaporation and under normal conditions will be well below freezing. Blowing a stream of air across the flasks or immersing them in cold water will increase the speed of evaporation.

The lyophilizer as shown is large enough to seal four $ joints around the cold finger in place of the three shown. Some operators prefer to have the joints sealed on at a slight angle instead of straight out as shown.

MIXING

Substances can react with each other only when they are in close contact; for this reason compounds containing different phases must be brought together to react and the products of this reaction moved in order to give unreacted molecules the opportunity to make contact.

When mixing is done by an agitator inside a container, the operation is called stirring, whereas if the whole container is agitated the process is called shaking.

The speed of chemical reactions depends on such conditions as temperature, solvent, efficiency of stirring or shaking, and stirring speed or shaking speed.

The efficiency of mixing which may be obtained from a given apparatus depends on the power input and the density as well as the interfacial tension of the phases being mixed. In some cases compounds may be added which reduce the interfacial tension (i.e., surface-active agents) and facilitate mixing operations. Figure 13 illustrates some of the many types of stirrers or impellers commonly used in laboratories, and which it will be necessary for a glassblower to make from time to time.

Laboratory experiments are usually carried out in round-bottomed flasks, Erlenmeyer flasks, or beakers. All of these are the least efficient containers possible for mixing, since the smooth sides allow the liquid to swirl rapidly around the flask instead of mixing thoroughly. The flask shown in Figure 14, described by Morton,[8] is an excellent type of glass container for mixing operations. The creases should be pressed inward in the sides of an ordinary three-necked flask. A 2-liter flask should have four vertical, equally spaced creases, each about 7.5 to 10 cm. long and 0.6 to 2.5 cm. deep, tapering in width from 0.3 to 3.75 cm. at the circumference. These creases act as vertical baffles, breaking much of the swirl and thereby creating excellent mixing.

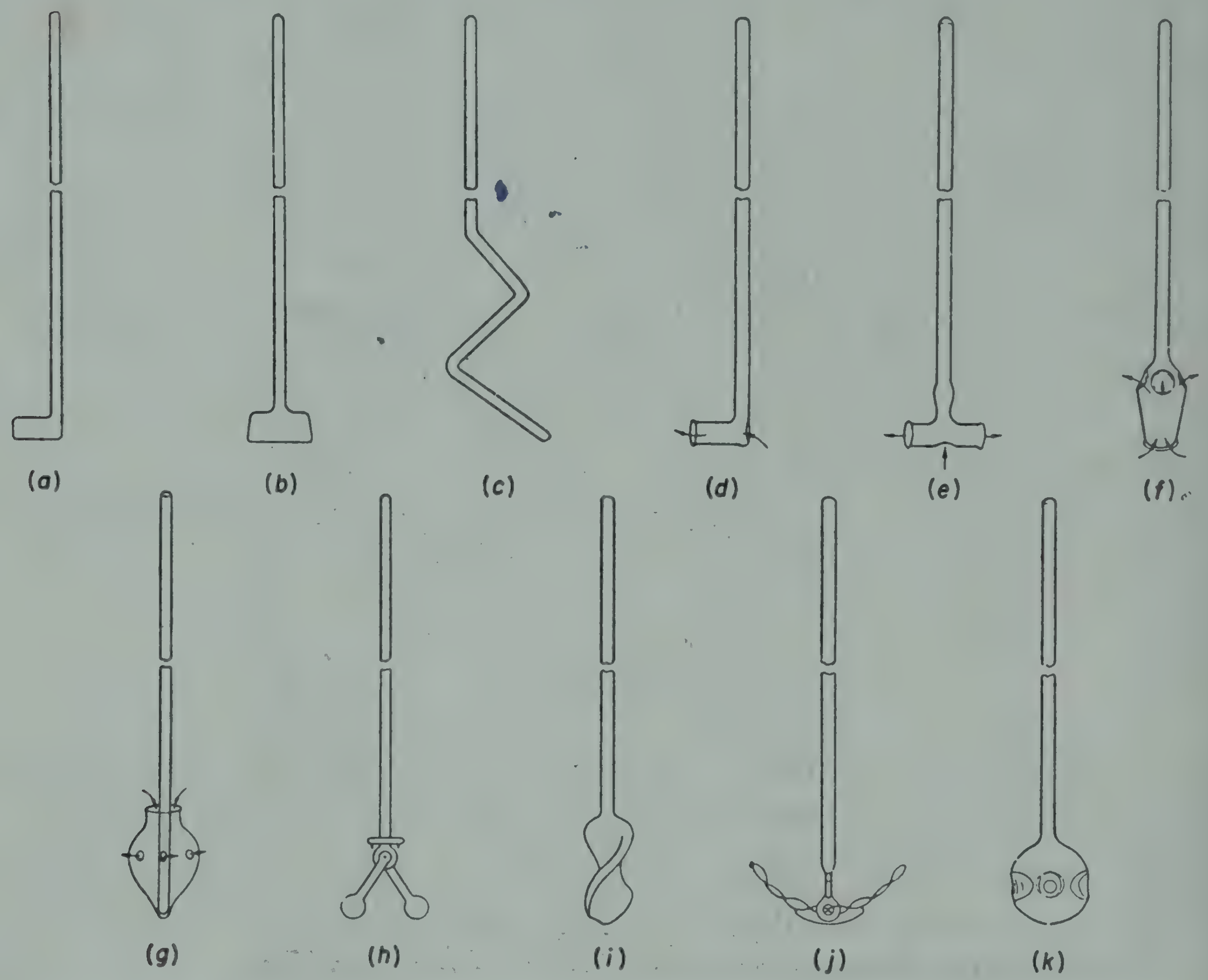

Figure 13. Laboratory stirrers and impellers. (From A. Weissberger, *Technique of Organic Chemistry*, Vol. III, 2nd ed., Part II.)

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[8] Morton, *Ind. Eng. Chem. Anal Ed.*, **11**, 170 (1939).

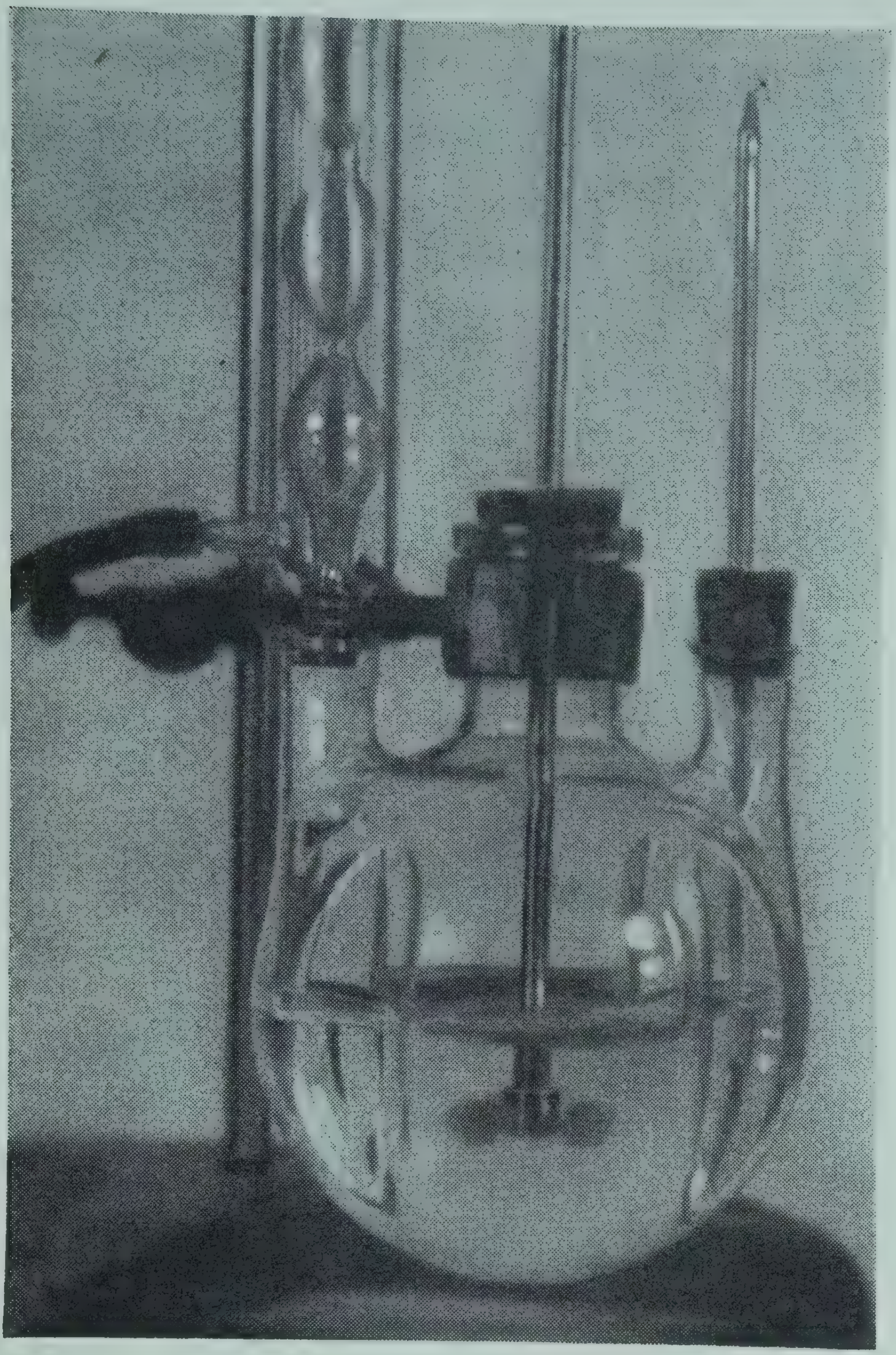

Figure 14. Morton stirring flask. (From A. Weissberger, *Technique of Organic Chemistry*, Vol. III, 2nd ed., Part II.)

SUBLIMATION

The sublimation of a solid substance is an unusual type of distillation process in which the solid vaporizes and the vapor is condensed directly into the solid state without the intermediate appearance of a liquid state. For sublimation to occur it is necessary for the solid to have a relatively high vapor pressure at a temperature below its melting point. Since, unfortunately, few organic compounds meet this condition, the technique of sublimation is not frequently used in general laboratory work. When it is applicable, sublimation affords the opportunity to obtain a product of high purity.

Sublimation is a slow operation but for small quantities of material (1 to 2 grams) it may be done at atmospheric pressure. A more satisfactory and certainly a quicker method of sublimation is that performed under reduced pressure. Figure 15A shows a simple apparatus for vacuum sublimation.

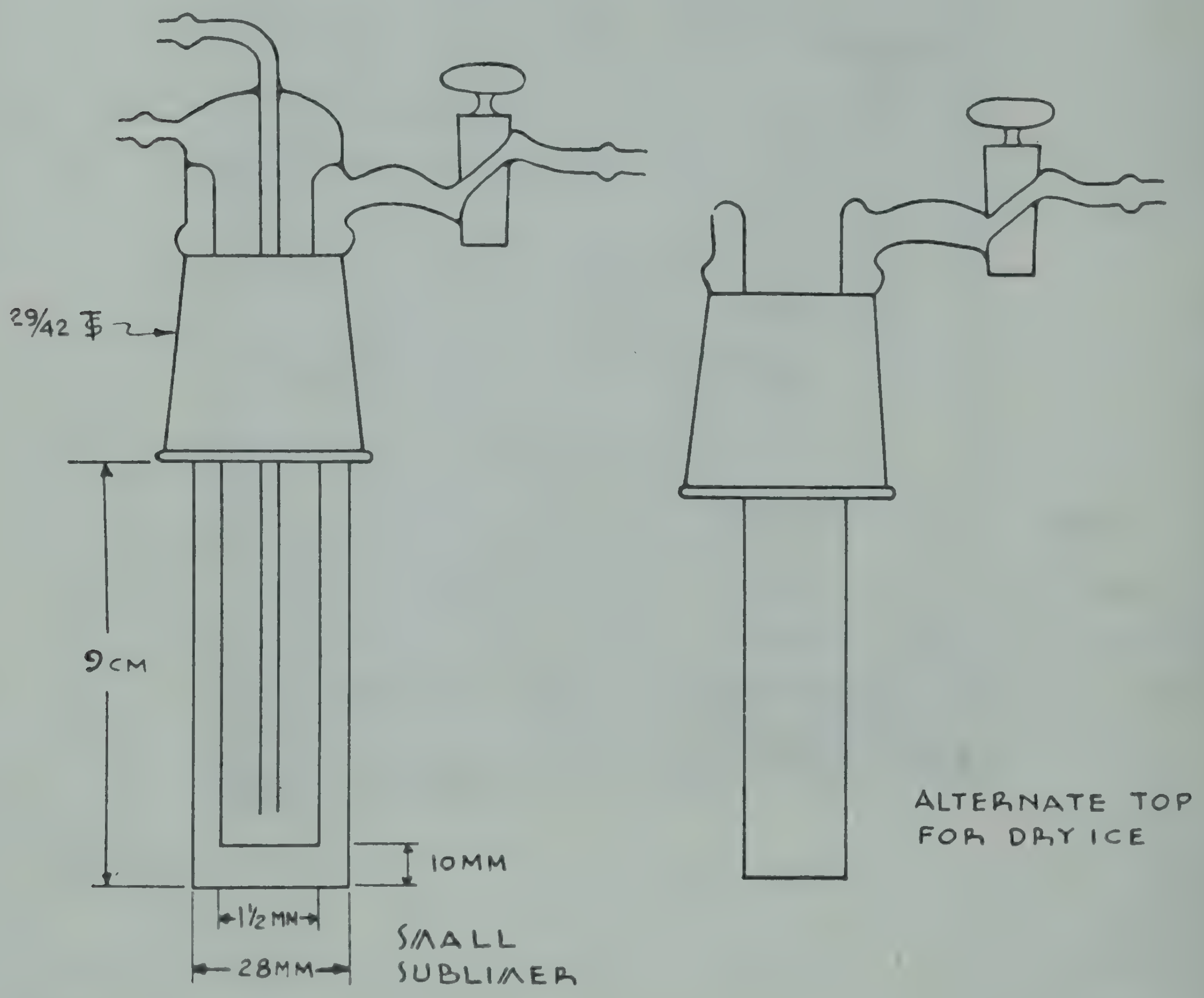

Figure 15A. Vacuum sublimator.

In making the sublimation it is important that the distance between the bottom of the cold finger and the outer or sample-holding tube be small. For the most successful sublimation this distance should not be greater than the mean free path of the molecules of the substance being sublimated, at the pressure being used.

In Figure 15B is shown a micro vacuum sublimator.

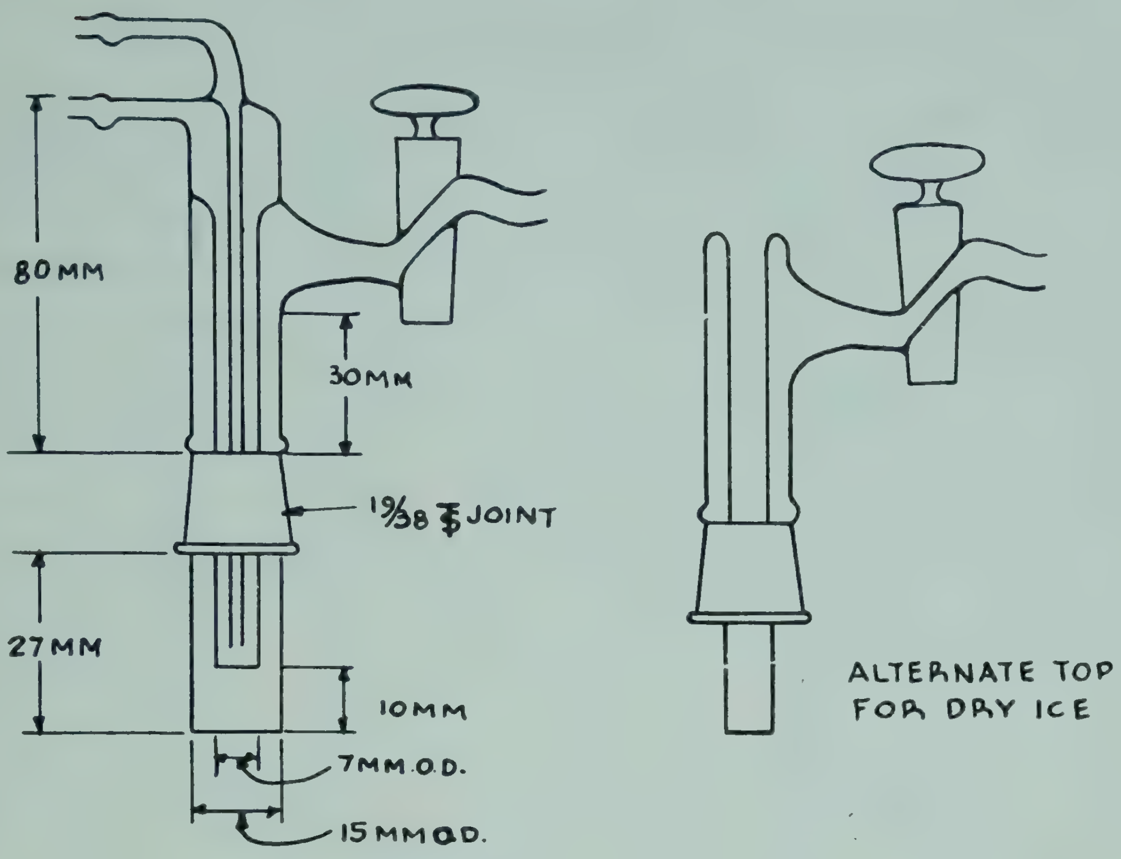

Figure 15B. Micro vacuum sublimator.

VISCOMETRY

The problem of determining the viscosity of a liquid and comparing this to the other physical properties of the liquid, as well as comparing the viscosity between various liquids, has been the subject of considerable research. The theory has been well covered by Weissberger.[9]

Viscosity is expressed in the following terms.

1. Absolute viscosity, in which the coefficient of viscosity of a liquid is defined as the force per unit area necessary to maintain a unit velocity gradient between two parallel planes.
2. Kinematic viscosity, in which the absolute viscosity is divided by the density of the liquid.

Liquids with viscosities ranging from 0.2 centipoise to 10^4 centipoises may be encountered, but most organic liquids have a viscosity in the range from 0.2 to 5 centipoises.

For work of this magnitude the capillary-type viscometers where the flow rate of the liquid is measured through a capillary under constant pressure are satisfactory. Several types of viscometers are shown in Figure 16.

[9] Weissberger, A., *Technique of Organic Chemistry,* Vol. 1, 2nd ed., Part 1. Interscience, New York, 1949.

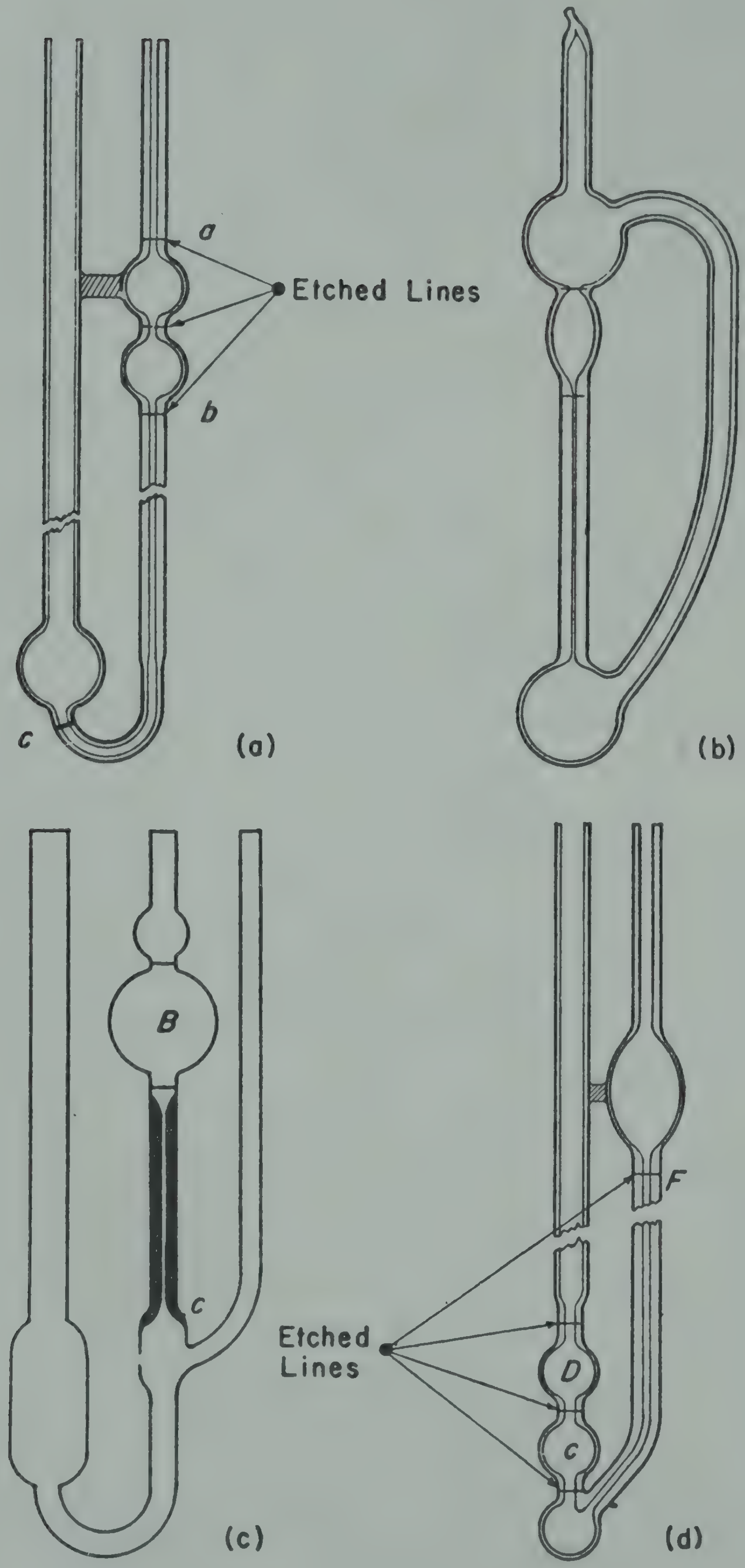
a
Etched Lines
b
c
(a)
(b)
B
c
(c)
F
Etched Lines
D
c
(d)

Figure 16. Viscometers. (From A. Weissberger, *Technique of Organic Chemistry*, Vol. I, 2nd ed., Part I.)

DENSITY

Density measurements are a convenient method for characterizing liquids. The most common method of determining density consists of finding the weight of liquid occupying a known volume at a known temperature. The temperatures most frequently used are 25/25° and 20/20° C., although references to other temperatures will be found in the literature. Vessels called pycnometers are used for making density determinations, and Figure 17 shows two designs and shapes of pycnometers. A pycnometer must be accurately calibrated, since the difference in weight when it is completely filled and when it is empty, together with the known volume of liquid, gives the density. The preliminary step to precise work with a pycnometer is the determination of its two volume constants, from which is derived the constant of the linear equation expressing the capacity as a function of the temperature. This should be done by filling the pycnometer with distilled water and accurately weighing it

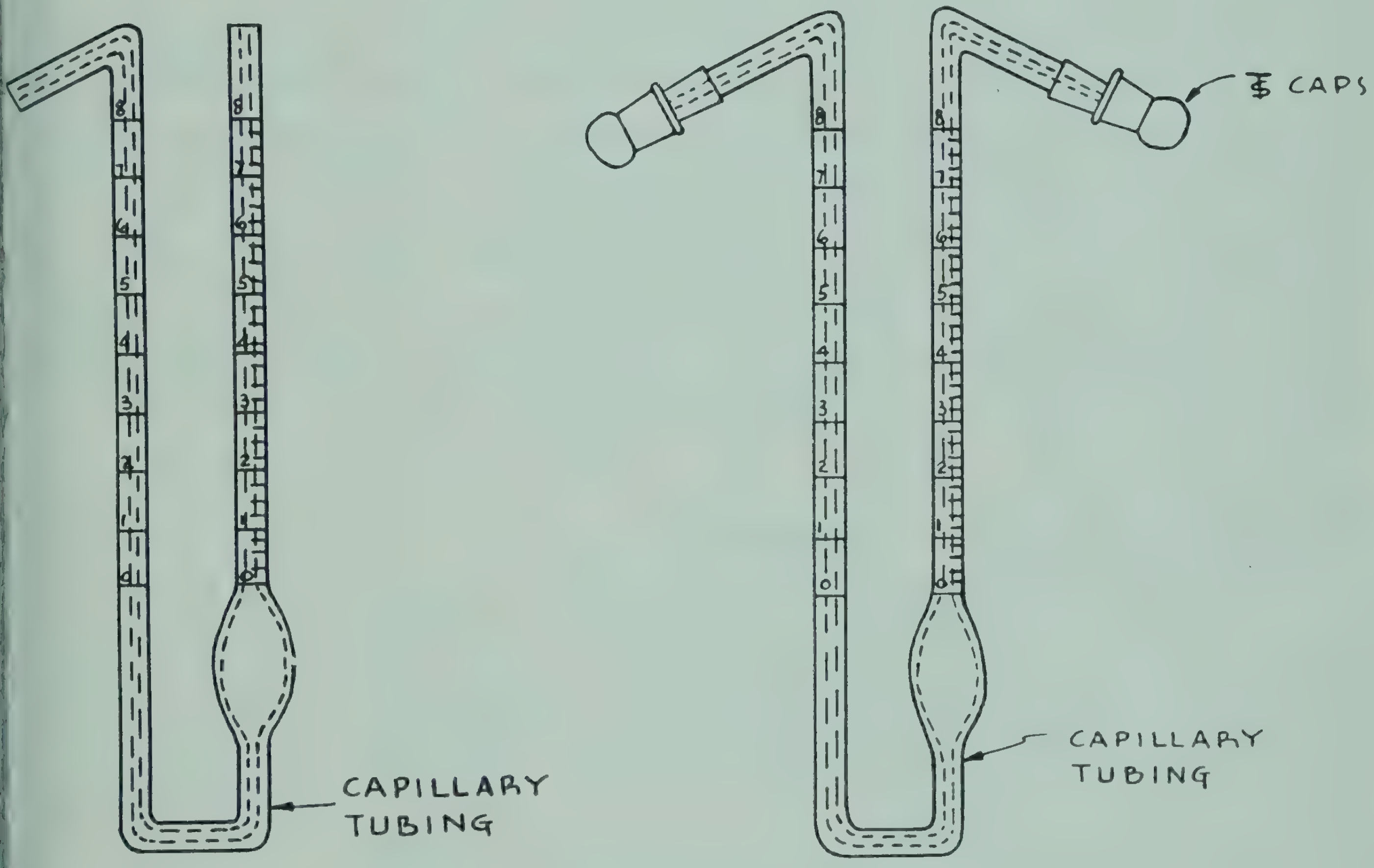

Figure 17. Pycnometers.

several times at each of the two temperatures near the end of the range for which the pycnometer is to be used. Pycnometers with $ caps over each end are preferable since the caps prevent contamination or loss of sample during weighing.

The density of solids is not a widely used constant. The many methods for determining this, however, are discussed by Bauer.[10] The most suitable method for routine work is probably the flotation method. The principle of this method is "A solid which remains suspended in a liquid, neither floating or sinking, has the same density as the liquid."

Reference mixtures which are used are bromoform, methylene iodide, carbon tetrachloride, benzene, nitrobenzene, and an aqueous solution of borium-mercuric iodide.

In practice the solid is added to the liquid in a centrifuge tube, and centrifuged at 2000 to 4000 r.p.m. for 5 minutes. If the solid settles, more of the lighter component is added. If the solid floats, more of the heavier liquid is added. This procedure is repeated until the suspended particles do not move appreciably, that is to say, have zero settling velocity when centrifuged, after which the density of the liquid is determined.

Removal of Frozen Stopcocks

1.	Alcohol	2 parts
	Glycerin	1 part
	Sodium chloride	1 part
2.	Chloral hydrate	10 parts
	Glycerine	5 parts
	Water	5 parts
	25 % Hydrochloric acid	3 parts

Either solution 1 or 2 may be used to free frozen joints and stopcocks. The solution should be poured on the stopcock or joint and allowed to stand until it becomes loosened. On occasion it may be necessary to allow the frozen members to soak overnight.

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[10] Bauer, "Determination of density" in *Physical Methods of Organic Chemistry*, Volume 1, Part 1, 2nd ed., p. 286. Interscience, New York, 1949.

Rust Preventive

Grinding wheels and other steel tools which are used around water can be protected from rusting by adding a small amount of sodium dichromate to the water solution.

Preparation of Ground Glass

The glass should be ground in any suitable grinding machine and screened through a set of standard screen sieves in which the various sizes are separated. A set which has screens of 10-20-30-40-50-60-80-100-150 and 200 mesh will be suitable for most grit of glass to be used.

After being ground the glass should be soaked in nitric acid overnight. This removes much of the impurity. After the nitric acid is filtered off, the glass should be given two or more thorough washes with distilled water. Hydrofluoric acid should then be stirred through the glass and allowed to stand for 15 to 30 minutes. After the hydrofluoric acid is filtered off, the ground glass should be thoroughly washed with several rinses of distilled water and dried in an oven at about 100° C. The thoroughness of the cleaning will be clearly visible after the drying. Rust-colored glass indicates that cleaning was not complete and the operation should be repeated. Glass which has been properly cleaned will have the appearance of white grains of sugar. Because of the wide variety and quality of commercial plates it is not generally necessary for the glassblower to make his own; however, special sintering can be done at between 770° and 880° C. for about 15 minutes.

Etching Glass

By Means of Glue. Glass may be etched by giving it a coating of ordinary glue dissolved in water. For a more positive action 6 % by weight of potash alum should be added while heating the mixture in a water bath. After the glue has melted and become perfectly homogenous and of the consistency of syrup, it should be applied to the glass while still hot. Application may be made with a brush and after one half hour a second coating should be applied over the first in such a way as to obtain a smooth surface without air bubbles. After the glue has become so hard that it can no longer be marked by the pressure of a fingernail, it should be placed in an oven in which the temperature is not allowed to rise above 105° F. When the glue becomes completely dried it will detach itself from the glass and in so doing will remove numerous scales of varying thickness, resulting in a decoratively designed surface.

By Means of Hydrofluoric Acid. Lines and figures may be etched on glass by coating the surface of the glass with beeswax, drawing the desired lines in the wax with a steel point, and exposing the glass to the fumes of hydrofluoric acid. Liquid hydrofluoric acid may be used but it is not always suitable since it leaves the surface transparent. After etching, transparent lines may be made more easily visible by being filled with a colored, commercial glass marking ink.

TABLE I. Conversion Factors

Quantity	To convert from	To	Multiply by
Speed	Liters/sec.	Cubic ft./min.	2.12
	Cubic ft./min.	Liters/sec.	0.472
Volume	Liters	Cubic ft.	0.0353
	Cubic ft.	Liters	28.3
Pressure	Atmospheres	Lb./in.^{2}	14.7
	Lb./in.^{2}	Atmospheres	0.068

TABLE II. Densities of Gases

	Density p_0 g.cm.$^{-3}$ at 0° C. 760 mm. Hg	Density p_1, g.cm.$^{-3}$ at 20° C., 1 dyne/cm.^{2}	$\sqrt{p_1}$
Air	1.293×10^{-3}	11.9×10^{-10}	3.45×10^{-5}
Hydrogen	0.090×10^{-3}	0.83×10^{-10}	0.91×10^{-5}
Helium	0.178×10^{-3}	1.64×10^{-10}	1.28×10^{-5}
Ammonia	0.771×10^{-3}	7.1×10^{-10}	2.66×10^{-5}
Carbon monoxide	1.250×10^{-3}	11.5×10^{-10}	3.39×10^{-5}
Nitrogen	1.257×10^{-3}	11.6×10^{-10}	3.40×10^{-5}
Oxygen	1.429×10^{-3}	13.1×10^{-10}	3.62×10^{-5}
Argon	1.784×10^{-3}	16.4×10^{-10}	4.05×10^{-5}
Carbon dioxide	1.974×10^{-3}	18.2×10^{-10}	4.26×10^{-5}

TABLE III. Mean Free Paths[1]

Gas	Temperature °C.	Mean free path at pressure		
		760 mm. Hg	1 mm. Hg	0.001 mm. Hg
Air	20	0.000,093 mm.	0.071	71.0
Hydrogen	20	0.000,17 mm.	0.13	130.0
Helium	20	0.000,27 mm.	0.205	205.0
Ammonia	20	0.000,065 mm.	0.050	50.0
Carbon monoxide	20	0.000,091 mm.	0.069	69.0
Nitrogen	20	0.000,092 mm.	0.070	70.0
Oxygen	20	0.000,098 mm.	0.074	74.0
Argon	20	0.000,097 mm.	0.074	74.0
Carbon dioxode	20	0.000,061 mm.	0.046	46.0
Water vapor	300	0.000,071 mm.	0.054	54.0
Mercury vapor	300	0.000,145 mm.	0.11	111.0

[1] This table is based on Dushman's calculated values, *Gen. Elec. Rev.*, **18**, 1045 (1915).

TABLE IV. Measure and Weights

1 Millimeter = 0.001 meter = 0.0394 in.
1 Centimeter = 0.01 meter = 0.3937 in.
1 Decimeter = 0.1 meter = 3.937 in.
1 Meter = 1.0 meter = 39.37 in.

1 Milliliter = 0.001 liter = 0.061 cubic in.
1 Centiliter = 0.01 liter = 0.6102 cubic in.
1 Deciliter = 0.1 liter = 6.1022 cubic in.
1 Liter = 1.0 liter = 0.9081 quart

TABLE V. Metric and English Equivalents

Millimeters × 25.4 = Inches
Meters × 3.2803 = Feet
Square millimeters × 1.09361 = Square inches
Square meters × 10.7641 = Square feet
Cubic centimeters × 16.3870 = Cubic inches
Ounces × 28.3495 = Grams
Pounds × 453.5926 = Grams

TABLE VI. Multiples

Diameter of a circle × 3.1416 = circumference.
Radius of a circle × 6.283185 = circumference.
Square of a radius of a circle × 3.1416 = Area.
Square of the circumference of a circle 0.07958 = area.
Square of the diameter of a circle × 0.7854 = area.
Half of the circumference of a circle × half its diameter = area.
Circumference of a circle × 0.159155 = radius.
Square root of the area of a circle × 0.56419 = radius.
Circumference of a circle × 0.31831 = diameter.
Square root of the area of a circle × 1.12838 = diameter.
Diameter of a circle × 0.86 = side of an inscribed equilateral.
Diameter of a circle × 0.7071 = side of an inscribed square.
Circumference of a circle × 0.225 = side of an inscribed square.
Circumference of a circle × 0.282 = side of an equal square.
Diameter of a circle × 0.8862 = side of an equal square.
Base of a triangle × one-half the altitude = area.
Multiply both diameter and 0.7854 together = area of an ellipse.
Surface of a sphere by one-sixth its diameter = solidity.
Circumference of a sphere × its diameter + surface.
Square of the diameter of a sphere × 3.1416 = surface.
Square of the circumference of a sphere × 0.3183 = surface.
Cube of the diameter of a sphere = solidity.
Cube of the circumference of a sphere × 0.16887 = solidity.
Radius of a sphere × 1.1547 = side of inscribed cube.
Square root of (one third of the square of) the diameter of a sphere = side of an inscribed cube.
Area of its base × one third of its altitude = solidity of a cone or pyramid, whether round, square, or triangular.
Area of one of its sides × 6 = surface of a cube.
Altitude of a trapezoid × one half the sum of its parallel sides = area.

TABLE VII. Pressure of Saturated Water Vapor

Temperature, ° C.	−110	−100	−89	−75	−60
Vapor pressure mm. Hg	7.5×10^{-7}	7.5×10^{-6}	7.5×10^{-5}	7.5×10^{-4}	7.3×10^{-3}
Temperature, ° C.	−50	−40	−30	−10	0
Vapor pressure[1] mm. Hg	0.029	0.096	0.783	1.96	4.58
Temperature, ° C.	10	20	30	40	50
Vapor pressure mm. Hg	9.21	17.55	31.86	55.40	92.6
Temperature, ° C.	60	70	80	90	100
Vapor pressure mm. Hg	150	234	355	526	760

[1] Figures denote pressure of saturated vapor above ice.

TABLE VIII. Vapor Pressures Relative to Brass

Material	Relative vapor pressure
Brass	1.0
Pyrex	0.7[1]
Iron	3.5
Rusty iron	25.0
Picein	3.5
Sealing Wax (Dennison Royal Scarlet)	4.5
Beeswax (1) and resin (1)	5.5
Sealing wax (Dennison No. 2)	12.0
De Khotinsky, hard	15.0
De Khotinsky, medium	21.5
De Khotinsky, soft	25.0
Glyptal, black	5.5
Glyptal, red	7.0
Glyptal, lacquer baked for 4 hours at 200° C.	2.0
Glyptal, lacquer dried in air for 14 days	5.0
N-di-butyl phthalate	5.4

[1] The vapor pressure of the Pyrex was due mainly to water vapor, since heating the glass for 10 minutes with a torch reduced the value from 0.7 to 0.06.

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